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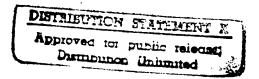
in

Materials for High Performance Applications

Final Proceedings of
The EOARD/IRC-sponsored
International Workshop on Gamma
Aluminide Alloy Technology

held from 1 to 3 May 1996 at The IRC in Materials for High Performance Applications The University of Birmingham

SECTION FOUR







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SECTION FOUR

19970620 019

The organisers wish to thank the United States Air Force European Office of Aerospace Research and Development for its contributions to the success of this conference

The Role of the Initial Steps of Oxidation for High Temperature Oxidation Resistance

C. Lang, M. Schütze Karl Winnacker - Institut DECHEMA D - 60061 Frankfurt / M.

1 Introduction

- insufficient oxidation resistance of γ titanium aluminides above 800°C.
- formation of a complex mixture of TiO₂ and Al₂O₃ instead of a thin, protective Al₂O₃ layer

gas / TiO_2 / (Al_2O_3) / TiO_2 + Al_2O_3 / Al depl. metal

- detrimental effect of nitrogen containing atmospheres
- niobium additions improve oxidation resistance
- investigation of the initial stages of oxidation

2 Experimental Procedure

materials: Ti36Al (mass - %)

Ti35Al5Nb (mass - %)



Experimental Procedure

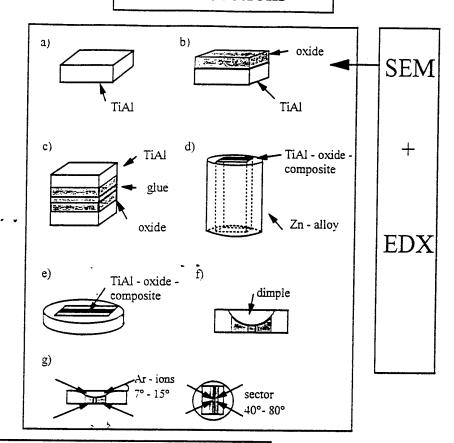
TEM sample preparation

foil samples

cross sections

• electrolytic jet thinning with the solution H2SO₄: CH3OH₂1:9 at -25°C

- subsequent short ion thinning for cleaning of the surface
- oxidation in air from 800°C to 1000°C



TEM investigation

energy dispersive X - ray analysis (EDX) — chemical composition

bright / dark field images

scale structure and morphology

selected area diffraction (SAD)

convergent - beam electron diffraction (CBED)

computer aided analysis of diffraction patterns

calculation of interplanar spacings d by the equation:

$$\lambda * L = R * d$$

d: interplanar spacing

R: distance of the reflex to the origin on the pattern

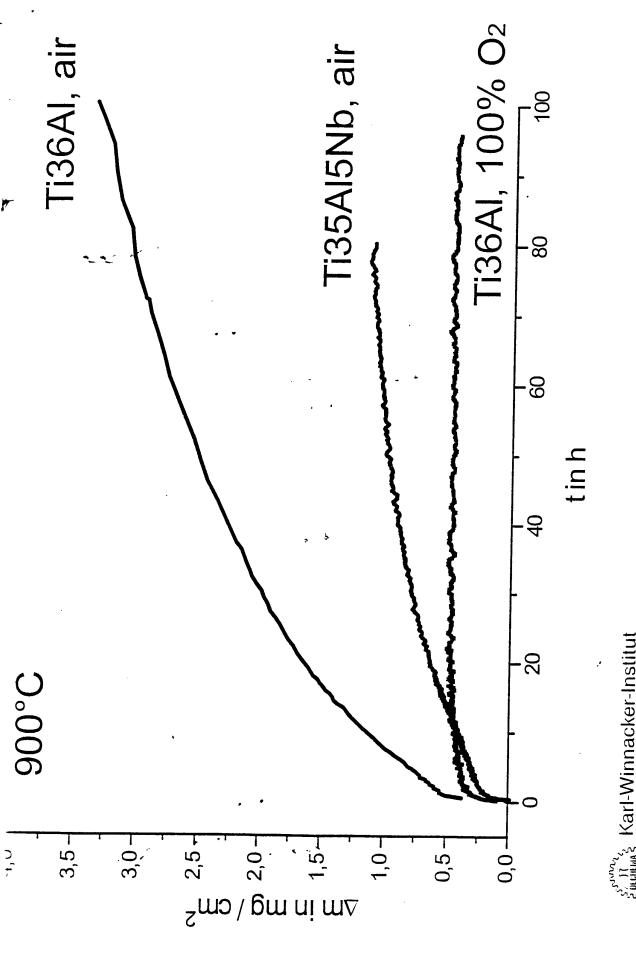
λ*L: camera constant

indexing of the diffraction patterns by the calculated interplanar spacings and identification of the phase

oxide scale structure, orientation relationship to the substrate

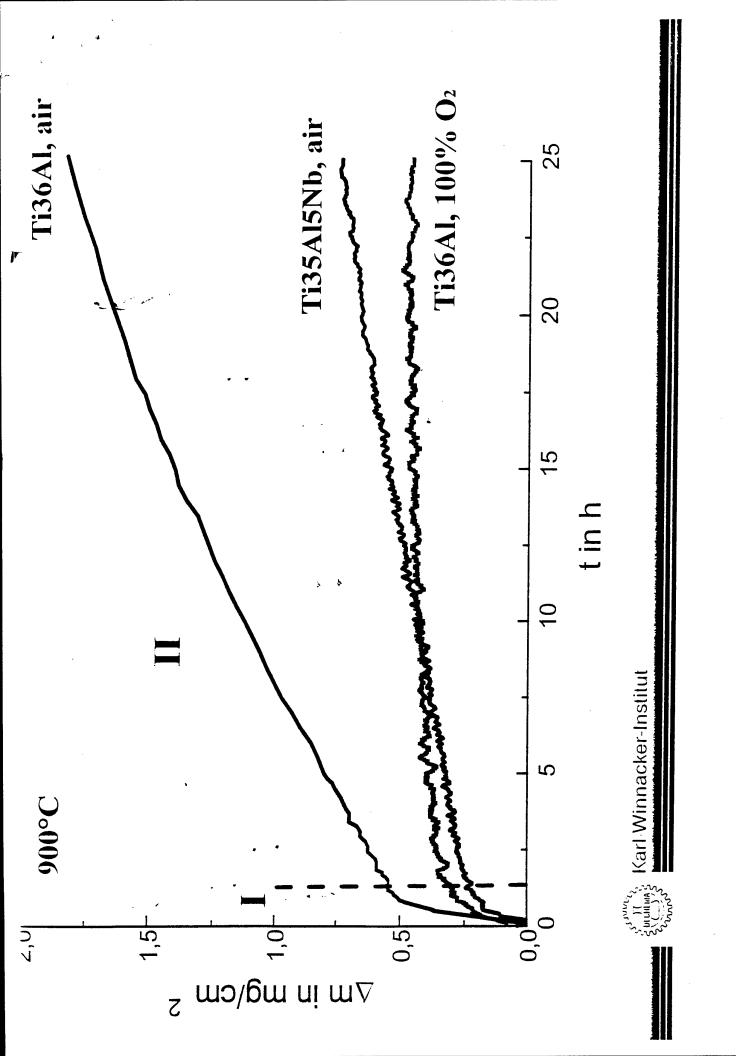


Karl-Winnacker-Institut

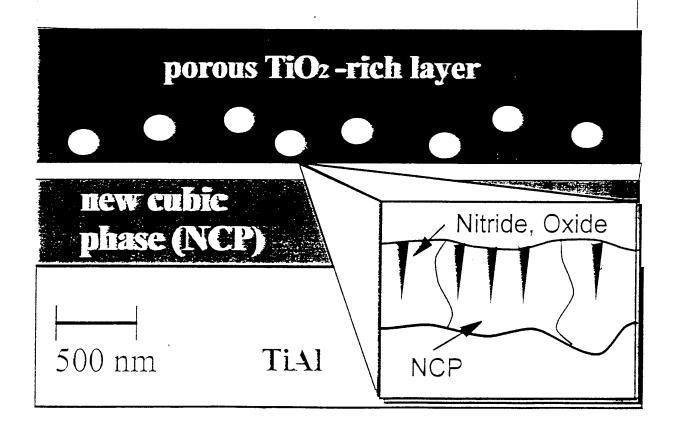




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oxide mixture Al₂O₃ + TiO₂



Schematic illustration of the oxide scale and the metal subsurface layer of Ti36Al after oxidation for 0.5 h at 900°C in air.





Metal subsurface zone of Ti36Al after 0.5 h of oxidation at 900°C in air.



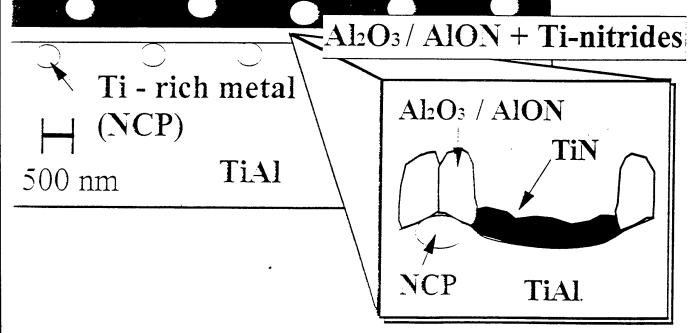
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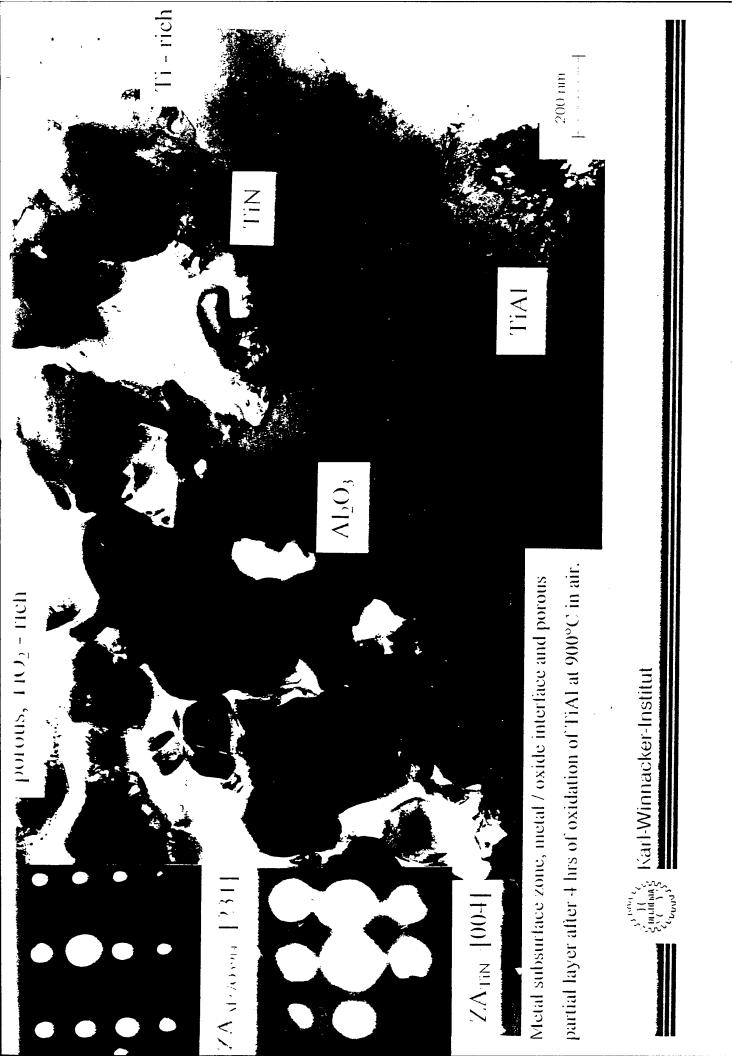
$$TiO_2 + Al_2O_3$$

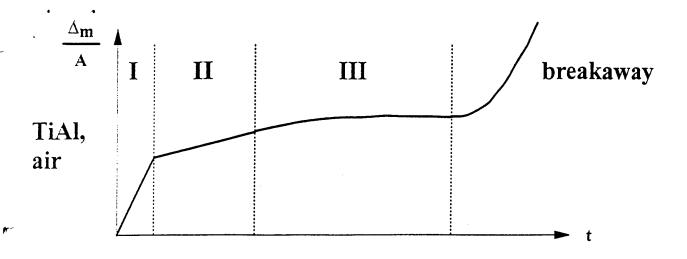
porous TiO2-rich layer



Schematic illustration of the oxide scale on Ti36Al after h of oxidation at 900°C in air.







I:

- initial formation of Al₂O₃ and TiO₂
- → Al depletion and subsequent for mation of Ti rich subsurface zone
- → increase of Ti activity and decrease of Al - activity
- → formation of Ti nitrides / oxides at the metal / oxide interface caused by the nonprotective oxide scale
- rapid oxide growth until consumption of the Ti - rich subsurface zone

II:

- change of the subsurface zone from Ti rich to TiAl
- → oxidation
 behaviour
 different to stage I
 because of the
 already formed
 oxide scale
- → linear oxide
 growth through
 repeated
 formation of Ti nitrides, Al₂O₃
 and Ti₃Al at the
 metal oxide
 interface

III:

- formation of the outer Al₂O₃ barrier
- parabolic oxide growth



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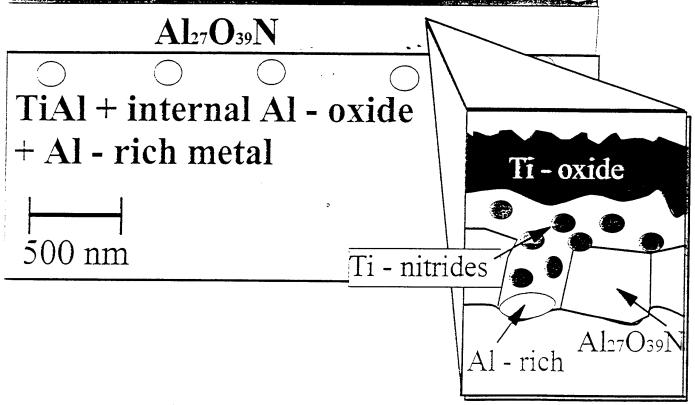
$\dot{t} = t_1$	oxide scale			
metal / oxide	metastable Al ₂ O ₃ AlON	s /	TiN	metastable Al ₂ O ₃ / AlON
interface	Ti-rich metal		TiAl	Ti-rich metal
$t = t_2$		Al Al ox:	idation of TiN to	d diffusion of
orevious nterface	pore	•	TiO ₂	pore
$t=t_3$	TïN		metastable Al ₂ O ₃ . AlON	TiN
	TiAl	,	Ti-rich metal	TiAl
	TiAl			
	oxide scale			
	pore		TiO2	pore
••••	TiO2		pore	TiO2
	metastable Al ₂ O ₃ AlON	/	TiN	metastable Al ₂ O ₃ / AlON
	Ti-rich metal		TiAl	Ti-rich metal
~~~~			TiAl	



# oxide mixture $\alpha - Al_2O_3 + TiO_2$



Ti - nitride + - oxide (porous)



Schematic illustration of the oxide scale on Ti35Al5Nb after 4 h of oxidation at 900°C in air





Metal oxide interface of Ti35Al5Nb after oxidation at 900°C for 4 h in air.

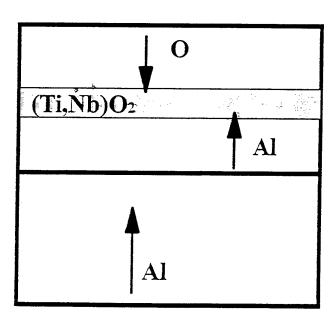
# • Effect of niobium addition

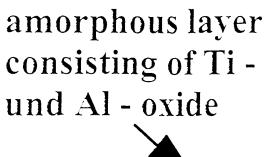
- no formation of an Al depleted metal subsurface zone
- stabilizing of a thin Al₂O₃ / AlON layer at the metal / oxide interface
- influence on ? TiAl phase field
- influence on the diffusion of Al and O in the metal and the oxide scale (doping of TiO₂)
- influence on the solubility of Al in TiO2

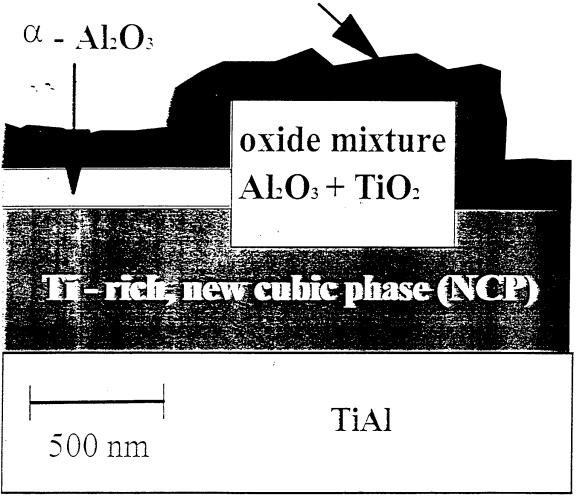
oxide scale

metal / oxide interface

**TiAl** 







Schematic illustration of the oxide scale and the metal subsurface layer of Ti36Al after oxidation at 900°C for 4 h in pure oxygen.



Oxide scale and metal subsurface layer on Ti36Al after 4 h of oxidation at 900°C in pure oxygen



Karl Winnacker-Institut

# Effect of nitrogen

- formation of Ti nitrides at the metal / oxide interface
- formation of metastable Al₂O₃ or AlON, respectively, at the metal / oxide interface instead of  $\alpha$  Al₂O₃

# Summary

- In nitrogen containing atmospheres metastable Al₂O₃ or AlON, respectively, is formed at the metal / oxide interface instead of  $\alpha$  Al₂O₃.
- In the metal / subsurface zone of Ti36Al Al depletion takes place and a new cubic phase (NCP) with a composition between α₂ Ti3Al and γ TiAl exists.
- During air exposure Ti nitrides are formed at the metal / oxide interface if an Al depletion occurs in the metal subsurface zone. Thus a continuous, protective Al₂O₃ layer is impeded.
- Niobium additions, which prevent Al depletion in the metal subsurface zone and stabilize Al2O3 / AlON formation at the interface, improve the oxidation resistance of ? titanium aluminides



# Development and Microstructural Assessment of TiAlbased Alloys at the IRC

Three general points concerning low alloy additions.

- (i) Additions of 2at% of elements such as Ta and W changed yield and UTS very little, but improved creep with respect to our base alloy of Ti 48Al 2Mn 2Nb.
- (ii) All other low alloying additions did little that could not be better accomplished by processing.
- (iii) Grain size refinement by B addition straightforward in base alloy but complicated in Ta and W-containing alloys.

# High Alloying additions

(i) Additions totalling about 8 - 10at% of elements such as Nb, Ta and Zr increase properties very signficantly. Life more complex with B2,  $\alpha$ 2 and  $\gamma$  phases and thermomechanical processing on these at early stage in IRC.

Property Targets Based on Industrial Specifications for LP Turbine Blades

Elongation  $\approx 2\%$ ;  $K_{1C} \approx 30 MPa \text{ m}^{0.5}$ Yield  $\approx 400 MPa$ ; UTS  $\approx 550 MPa$ 

Alloys/processing based on derivatives of Ti45Al2Nb2Mn, ie. low alloying additions to Ti45Al

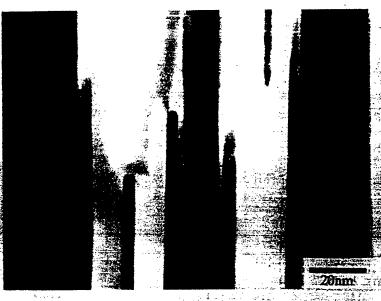
Property Targets Based on Industrial Specifications for HP Compressor Blades

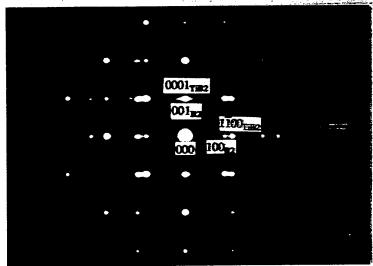
Elongation 3%; K_{1c} 30MPa m^{0.5}; Yield 700MPa UTS 1000MPa

Alloys/processing based on derivatives of Ti45Al with alloying additions of ≈ 8at% to Ti45Al

In neither case was a creep specification forthcoming! 1000h rupture tests at two temperatures and two stress levels are used to define relative primary and secondary creep strains

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 $\{1100\}_{\text{TiB2}} / \{100\}_{\text{B2}} \text{ and } [0001]_{\text{TiB2}} / \langle 001\rangle_{\text{B2}}$ 

a=303pm TiB2

cubic **B2** 

a=322pm

hex

c=322pm



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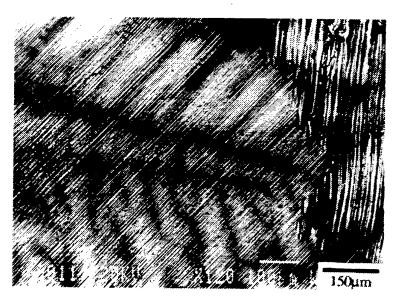


# The Effect of Non-Turbulent Solidification



Ti-47AI-2Cr-2Nb

Ti-47Al-2Cr-2Nb-1B



BSEI of Ti-47AI-2Cr-2Nb-1B

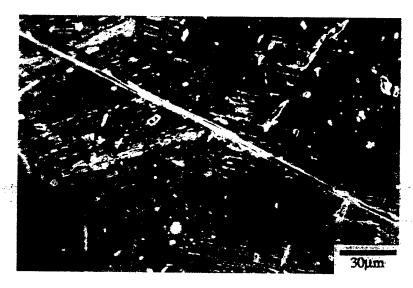


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Boride morphology in Ti-47Al-2Cr-2Nb-1B



Boride morphology in Ti-47Al-2Ta-1Mn-1Cr-1B-0.2Si.



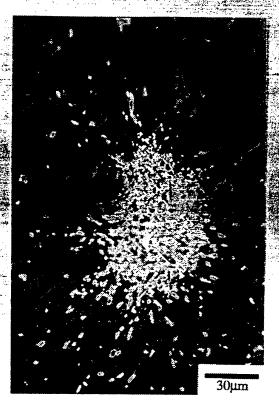
IRC in Materials for High Performance Applications



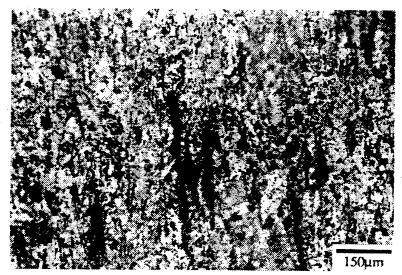
# Boride Agglomerates in Ti-47Al-2Ta-1Cr-1Mn-1B-0.2Si



As-cast — Bright field



As-cast — Dark field

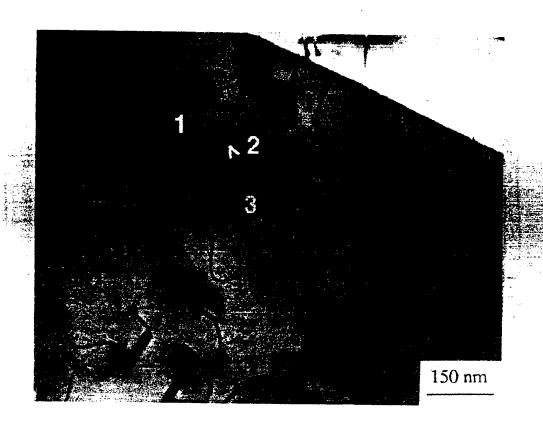


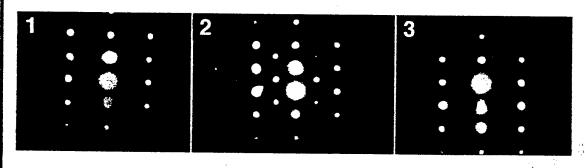
As-forged — forging axis



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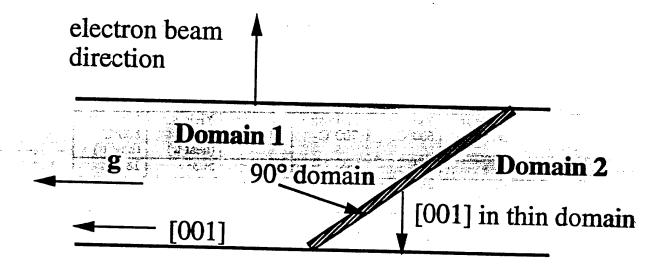


Typical APDBs in quenched Ti - 49Al and diffraction patterns from regions 1 - 3

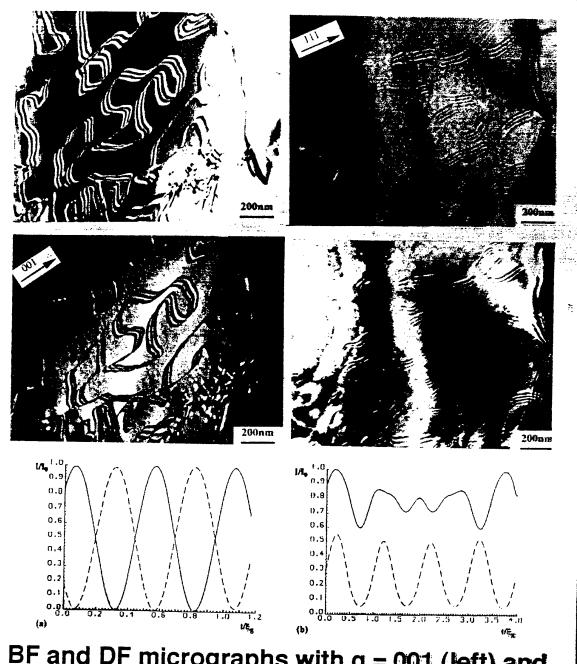


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1/2 <101> displacement between domains 1 and 2



BF and DF micrographs with g = 001 (left) and g = 111 (right) each at Bragg. Computed images assume  $\Delta R$  of 0.05 and appropriate deviations from Bragg for 90° domain



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HREM of APDB in Ti-48Al-0.5Mo



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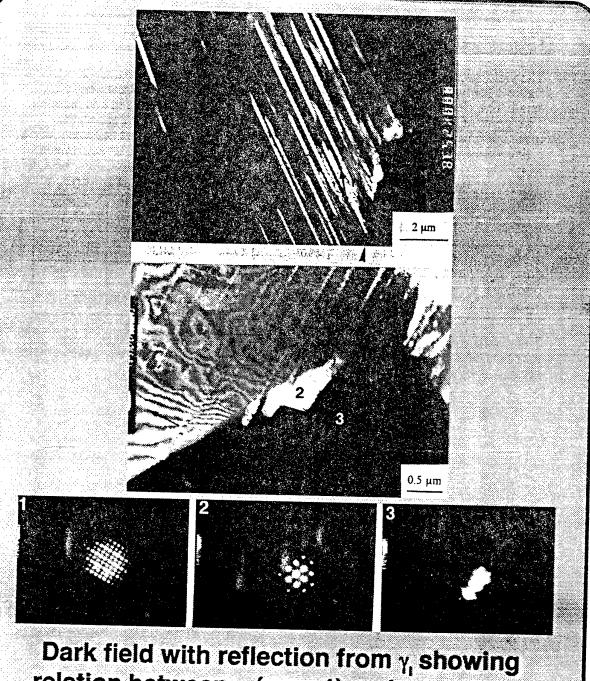






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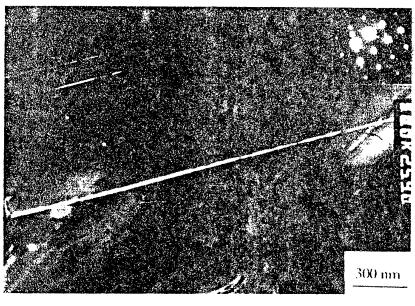
Dark field with reflection from  $\gamma_i$  showing relation between  $\gamma_i$  (area 1) and massive  $\gamma_m$  (area 2) and absence of relation between  $\alpha$  (area 3) and  $\gamma_m$ 

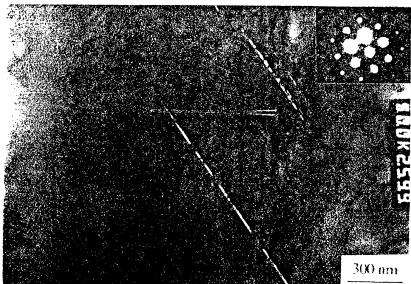


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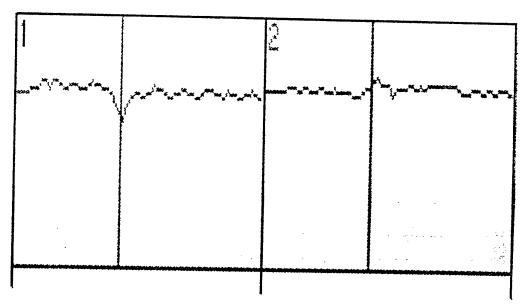


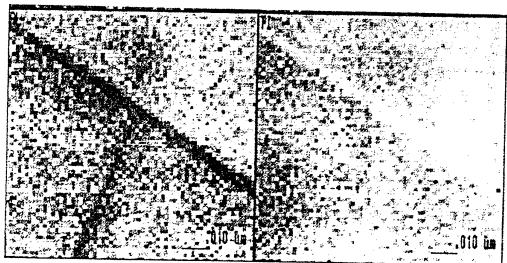
Dark field images of thin  $\gamma$  and thin  $\alpha \text{2}$  in  $\gamma_{\text{m}}$ 



IRC in Materials for High Performance Applications







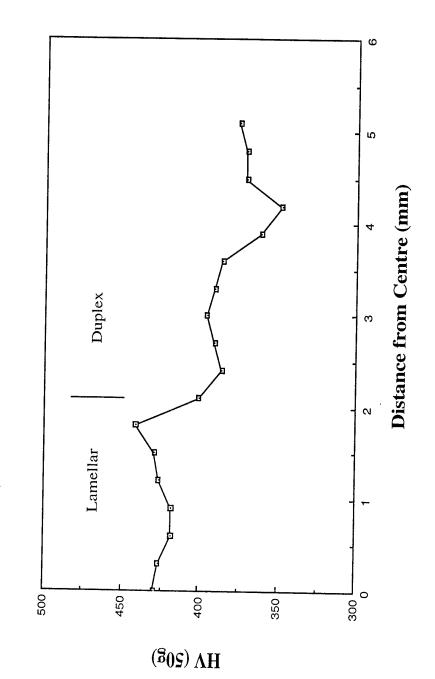
EDX trace and map showing AI depletion across thin  $\alpha$ 2



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Hardness traverse across sample extruded at 1380°C reduction in area 8.5 (Ti 48Al 2Cr 2Nb 1B)



# Recent Activities and Future Directions in Gamma Titanium Aluminides in the Study of Microstructures

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29, Av. de la Division Leclerc, 92322 Châtillon CEDEX, France

University of Orsay (R. Penelle, T. Baudin, M.L. Dupont) Collaborations: CEMES-CNRS of Toulouse (A. Couret, S. Zghal)



# Numerous issues

- 1) Case of two-phase  $\gamma + \alpha_2$  alloys such as the GE one
- solidification texture
- by heat treatments or by thermomechanical processings? - is it possible to reduce or suppress this texture
- new types of transformation modes
- Widmannstätten microstructure, "feather-like" microstructure
- is it possible to take advantage of these transformation modes?
- · quantitative analysis of the lamellar structure
- information on the distribution of orientation variants -> texture on a microscopic scale?
- 2) Case of three-phase  $\gamma + \alpha_2 + \beta$  alloys (very good creep properties with reasonable tensile ductility)
- reduction of solidification texture
- more information required both on phase diagrams and on phase transformations
- thermal stability of the  $\beta$  phase
- observed β phase: residual or in equilibrium?
- to phase precipitation

# Solidification text

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. 12

### **[exture**]

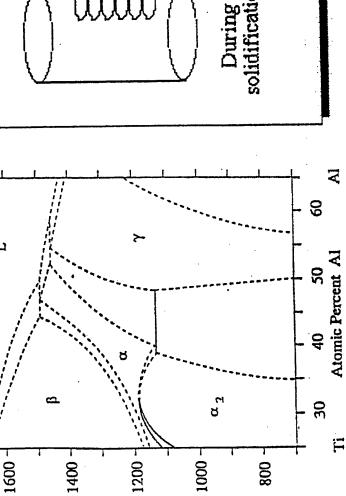
During solidification

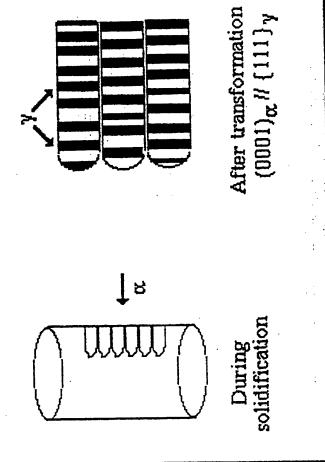
$$\Gamma \Rightarrow \alpha$$

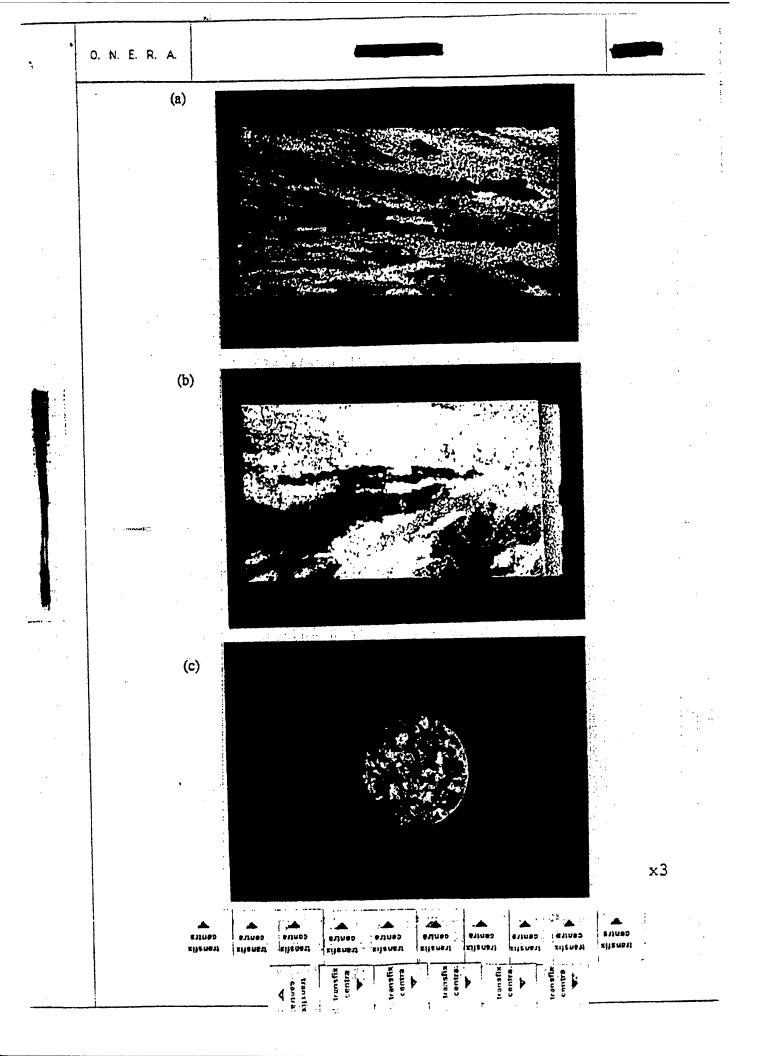
 $\alpha: HCP \Rightarrow crystal growth direction // [0001]$ 

During the formation of lamellar structure

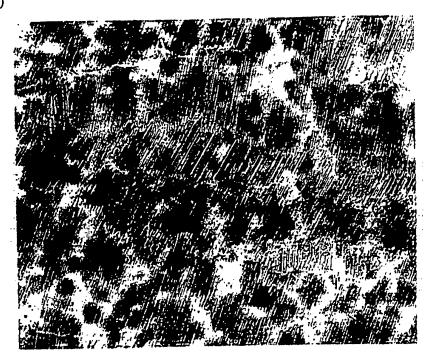
$$\alpha \Rightarrow \alpha + \gamma \Rightarrow \alpha_2 + \gamma \text{ (or } \alpha \Rightarrow \alpha_2 \Rightarrow \alpha_2 + \gamma)$$
  
(0001) $\alpha \text{ or } \alpha_2 / / \{1111\}\gamma$ 





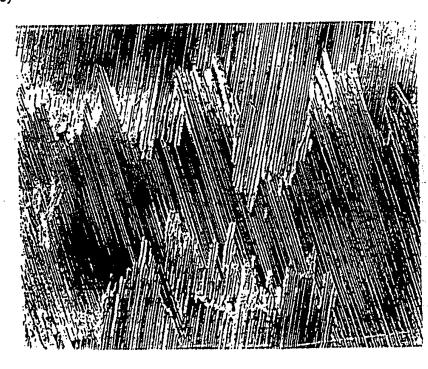


(a)



x50

(b)



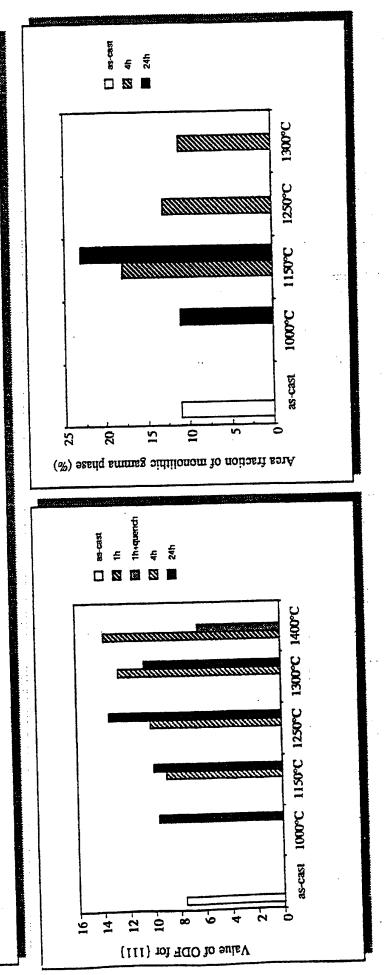
x200

sanata vanetix transfit transf

# Evolution of the texture during heat treatments

Collaboration with University of Orsay

• Texture analyses using neutron diffraction and SEM-EBSD show that the solidification texture cannot be reduced through heat treatments: the 111 (parallel to the axis of the rod cut from the ingot and following its radial direction) texture is even reinforced in many cases.





# Thermomechanical processings of strongly textured alloy ingots

### · Forging

- Forging is not difficult to conduct but deformation may be heterogeneous.
- recrystallized grains alternating with non recrystallized lamellar - Forged products show a banded structure.
  - Crystallographically, the texture is not suppressed but modified.

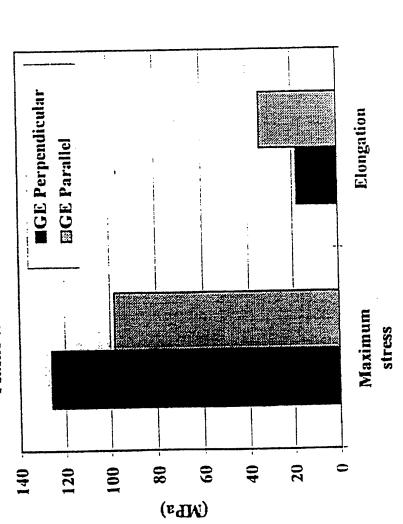
### • Extrusion

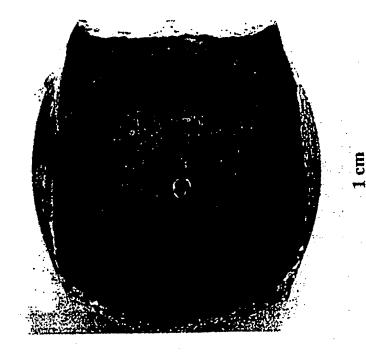
- Extrusion is difficult partly because of the orientation of columar grains.
- Is the route (forging + extrusion + forging) economically viable?!!! Industrial processing route: extrusion + forging



## High temperature deformability





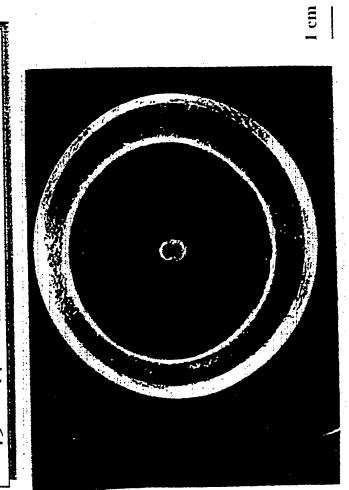




### Shape of forged pancakes

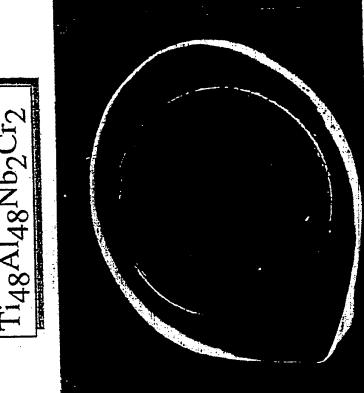
Forging conditions: 1150°C and  $5 \times 10^{-3} \text{ s}^{-1}$ 

 $Ti_{49}AI_{47}Nb_2Mn_2 + 2.4vol.\%TiB_2$ 



homogeneous deformation

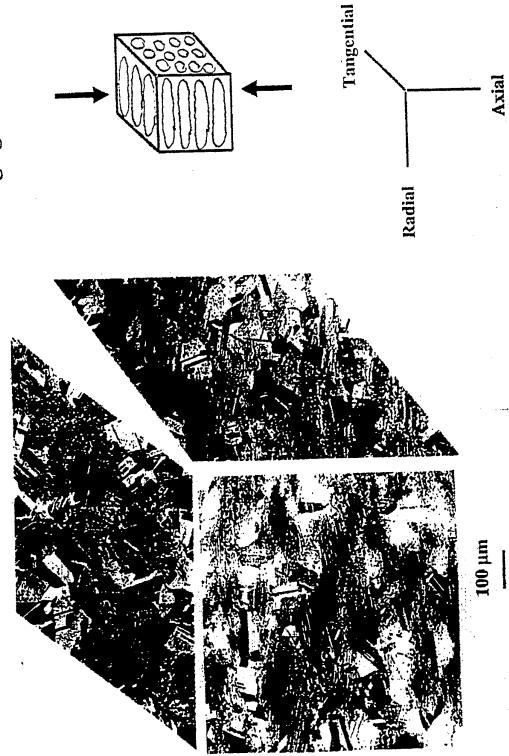
Ti48Al48Nb2Cr2



Heterogeneous deformation due to solidification texture

## As-forged microstructure

Forging at 1150°C - 80%



# New transformation modes

## Various transformation modes

• Formation of lamellar structure

- 
$$\alpha \Rightarrow \alpha + \gamma \Rightarrow \alpha_2 + \gamma \text{ or } \alpha \Rightarrow \alpha_2 \Rightarrow \alpha_2 + \gamma$$

- nature of various interfaces:

$$\gamma/\alpha_2$$
 and  $\gamma/\gamma$  (TB's, ODB's, PTB's, MB's)

Massive-type transformation

$$-\alpha \Rightarrow \gamma$$

- numerous defects and interfaces including  $\gamma$  APB's

 $\bullet$  Discontinuous coarsening of  $\gamma$  lamellae

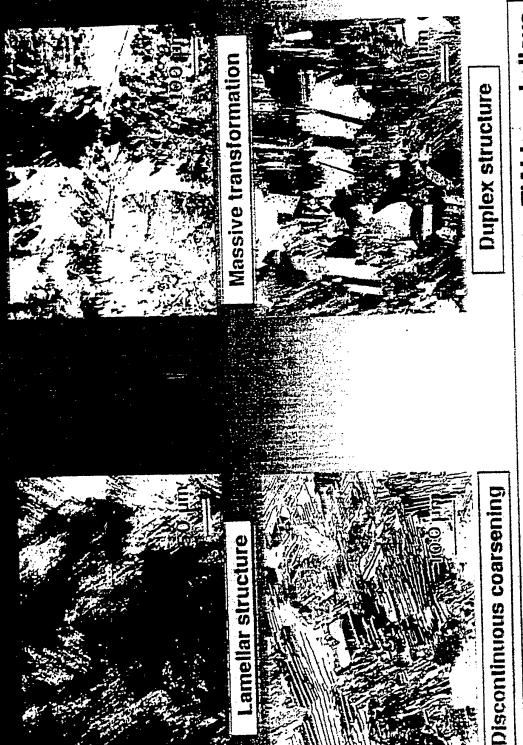
- solute redistribution through moving grain boundaries?

- mechanism less clarified

• Formation of "Widmannstätten" structure

• Formation of "feathery structure"

一番をからいる



Various microstructures observed in two-phase TiAl-based alloys



## New types of transformation mode

when heat treatments are conducted at temperatures around the  $\alpha$  transus (both in the  $\alpha$ -phase • These new transformation modes (Widmannstätten and feather-like) can be observed field and in the upper part of the  $\alpha+\gamma$  phase field).

### Widmannstätten structure

- observed after slow cooling (eg. furnace cooling) in the alloys initially showing a lamellar structure
  - most frequently observed when the alloy has a near  $\gamma$  composition.
- presence, in a lamellar grain, of zones where the orientation of lamellae is different from that of the matrix
  - formation mechanism probably related to  $\gamma \rightarrow \alpha$  transition upon heating

### • Feather-like structure

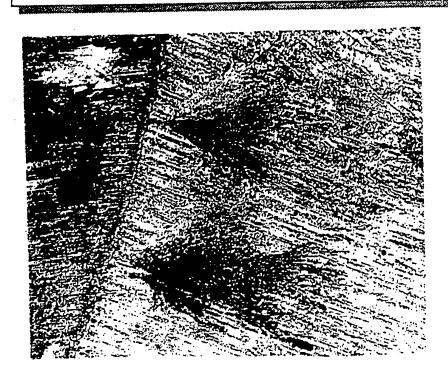
- observed after higher-rate cooling (eg. air cooling), but not after highest-rate cooling (eg. oil quenching)
  - deviation from the normal lamellar structure?
- formation mechanism less clarified and many further careful experiments required
- Possibility of benefiting the occurrence of these transformation modes in order to obtain the lamellar structure with a small grain size through appropriate heat treatments?

### Widmannstätten structure Structure "Widmanstätten"



 $200 \mu \text{m}$ 

Feather-like structure
Structure "Plumeaux"



 $100 \mu m \,$ 

### WIDMANNSTATTEN PLATES.



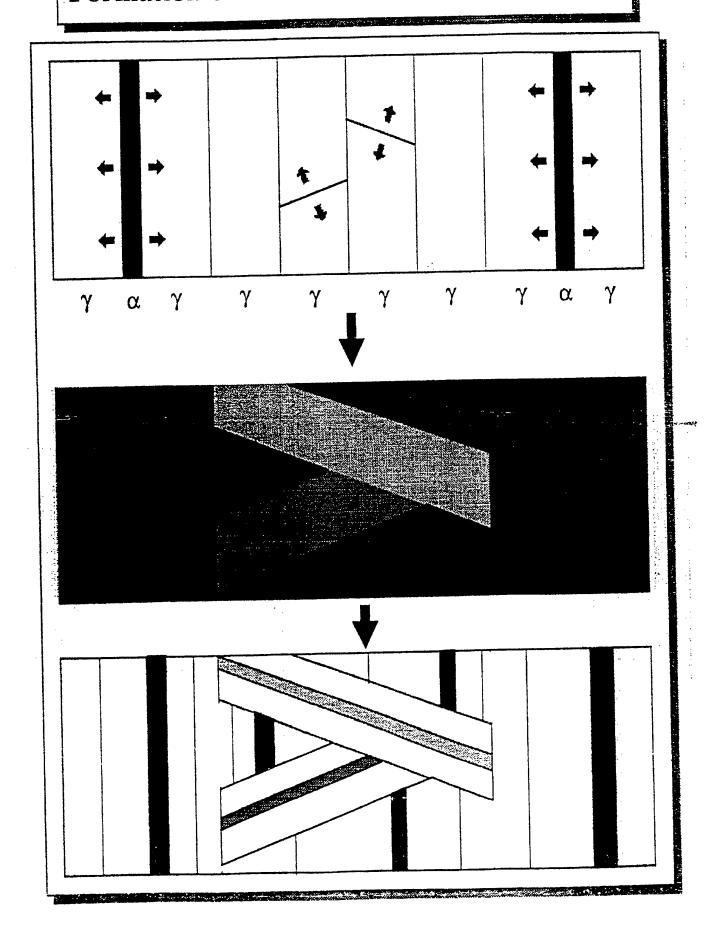
1,5 µm

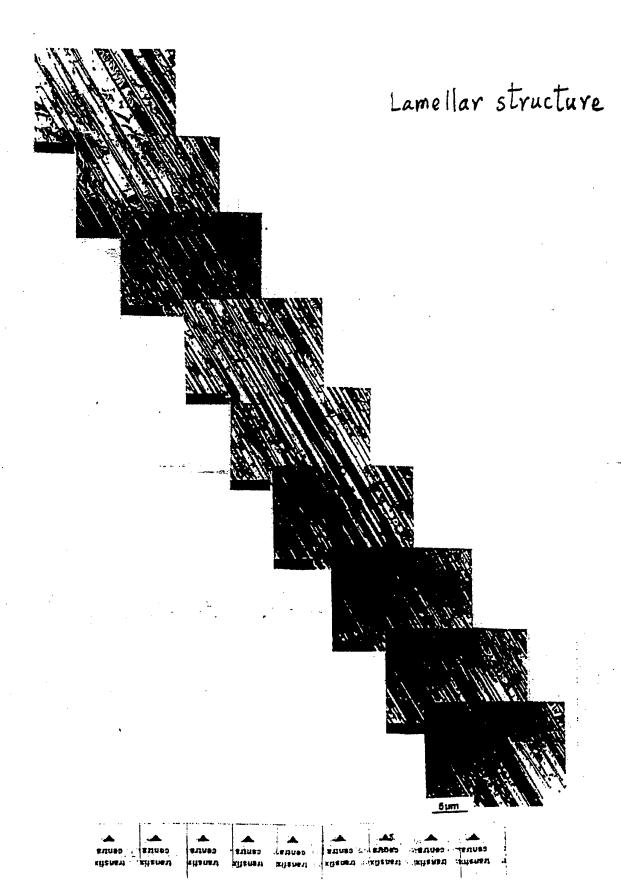
Ti₄₈Al₄₈Cr₂Nb₂.

1400°C/1h/ furnace cooling.

ONERA

### Formation of the Widmannstätten structure



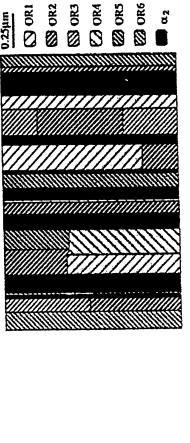


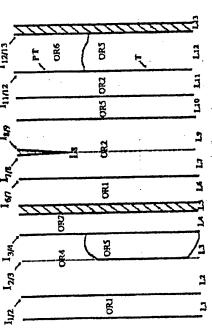
### of the lamellar structure Quantitative analysis

# Quantitative analysis of the lamellar structure

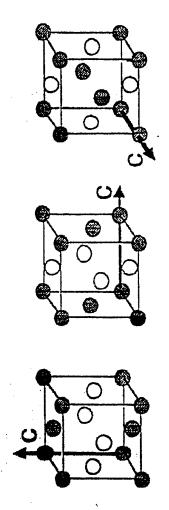
Collaboration with CEMES-CNRS, Toulouse

- Experiments
- a PST Ti_{50.7}Al_{49.3} crystal (provided by Prof. Yamaguchi) - specimens: an as-cast polycrystalline  ${\rm Ti}_{54}{\rm Al}_{46}$  alloy and
  - TEM: diffraction analyses and dark field imaging
- in the Ti₅₄Al₄₆ alloy; an area of 6.4 $\mu$ m by 9.5 $\mu$ m including 116 lamellae ( $\gamma$ : 77 and  $\alpha_2$ : 39)
  - in the PST alloy: several areas of about 20µm by 30µm
    - Microstructural parameters examined
- $\gamma$  and  $\alpha_2$  lamellae, six  $\gamma$  orientation variants in number and in volume fraction
  - different lamellar interfaces in number and in area fraction



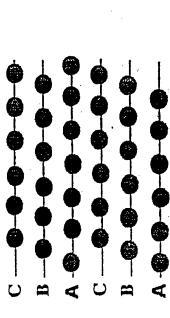


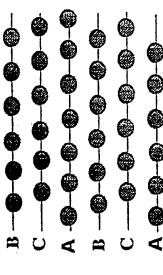
# Orientations of the $\gamma$ domaines in the lamellar structure



1) non-equivalence of the three <100> axes in  $\gamma$  (L10)

## ⇒ Existence of three variants of orientation

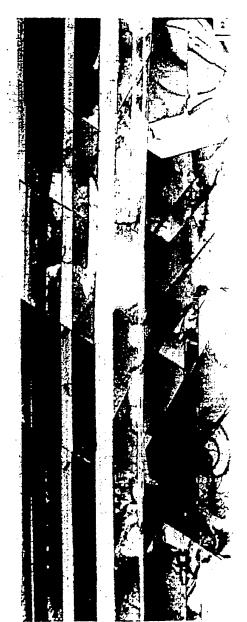




2) Possibility of two stacking sequences for {111} planes of the L1₀ structure, ordered on FCC lattice: ABCABC... or ACBACB...

 $\Rightarrow$  Six (3 X 2) possible orientations of the  $\gamma$  phase





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### Numerous very interesting findings

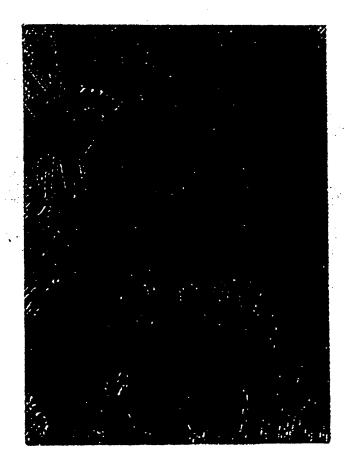
- while 63% of  $\alpha_2$  lamellae are bordered with two  $\gamma$  lamellae of the same stacking sequence. • In the Ti54Al46 alloy, 71% of \psi interfaces are twin or psedo-twin boundaries,
- $\Rightarrow$  During the lamellar structure formation, joining of  $\gamma$  lamellae of the same stacking sequence is more difficult than that of  $\gamma$  lamellae of the opposite stacking sequence due to a repulsive force between two ledge fronts.
  - $\Rightarrow$  The fact that some interfaces are separating two  $\gamma$  lamellae order-domain related, while orderdomain boundaries inside a lamella is wavy, may be explained with a hypothesis that a small number of aomic planes corresponding to the  $\alpha_2$  phase subsist between the two  $\gamma$  lamellae.
    - In the two alloys, twin boundaries are more frequently observed than pseudo-twin boundaries both in number and in area fraction, while, theoretically, the latter should form two times more frequently than the former.
- ⇒ minimization of mismatch energy

### Future experiments

- GE alloy before and after heat treatments
- Possible existence of micro(or local)-texture

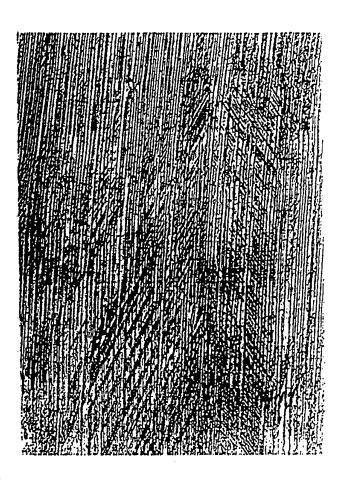


### Twining during creep



50 mm

Twining in monolithic y grains



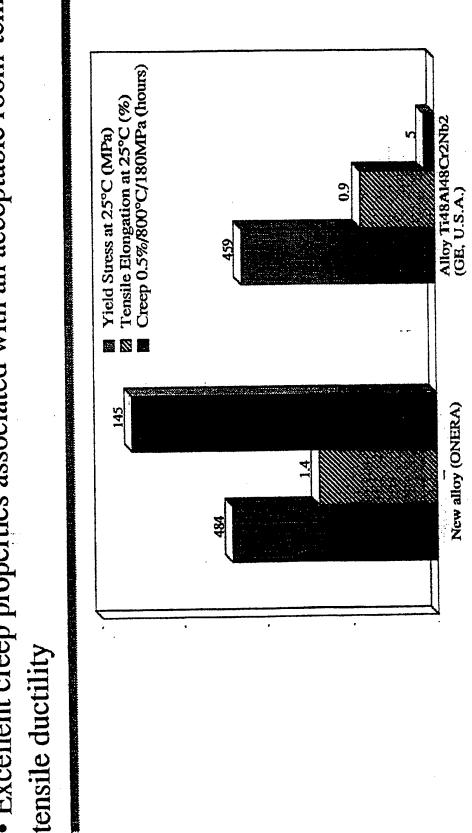
 $50 \, \mu m$ 

Twining in lamellar grains

# Three-phase $\gamma + \alpha_2 + \beta$ alloys

## Alloys appropriate to casting

- $\rightarrow$  development of new three-phase ( $\gamma + \alpha_2 + \beta$ ) alloys · Detailed examinations of the role of solidification paths
- Excellent creep properties associated with an acceptable room-temperature



### Conclusions

- but also in order to garantee or improve the minimum value for various not only in order to develop new alloys of higher-performance mechanical properties by identifying and controlling pertinent microstructural parameters during processing. Study of microstructures is very important
- For doing so, it is essential to combine various experimental techniques:
- e.g. optical microscopy,
- SEM including EBSD
- X-ray and neutron diffractions
- TEM including high resolution lattice imaging
- atom-probe experiments

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### Gamma Titanium Aluminide Alloy Technology: Status and Future

Young-Won Kim UES, Inc., Dayton, OH 45432 (April, 1996)

Gamma alloys are emerging as revolutionary engineering materials for high temperature structural applications. This article discusses the historical background, status and future prospect of gamma alloy technology in the areas of fundamental understanding, alloy development/design, process development, and applications.

### Introduction

Since the first measurements of mechanical properties and oxidation resistance in a binary TiAl cast alloy made in the early 1950's, numerous reports have confirmed many properties, beneficial to high temperature structural applications, including low density, high temperature strength retention, high stiffness especially at high temperatures, thermal expansion comparable to current alloys, good oxidation resistance, and hot corrosion resistance comparable to, or better than, those of current alloys. [1-15]

The first major gamma alloy development program was initiated by Air Force Materials Laboratory and conducted by Pratt and Whitney from 1975-1983. This exploratory program evaluated numerous alloy compositions through wrought processing and recommended Ti-48Al-1V-(0.1C) as the best alloy composition on the basis of ductility and creep resistance. Nevertheless, it's properties in the fine duplex microstructure condition were not adequate for the requirements of any engine components. At the end of the program, alloy castings were also evaluated; however, the properties were found to be unsatisfactory as the large-grained cast lamellar microstructures resulted in poor ductility and low strength. A few years later, the second major development program initiated again by the Air Force was performed by General Electric, Schenectady from 1986-1991. Largely based on the knowledge accumulated during the first program effort and other independent investigations, the effort identified Ti-48Al-2(Cr or Mn)-2Nb as the best second-generation alloy composition. The alloys, produced through the rapid-solidification/wrought processing, had a fine duplex microstructure and exhibited ductility/strength and oxidation resistance improved over those of the first-generation gamma alloys. Understanding the effects of alloying elements as well as composition on the properties progressed for both binary compositions and multicomponent alloy systems [6-10].

As in most metallic materials, investment casting was used as the first process route for producing experimental gamma components [6-10, 12, 14]. Since Howmet initiated a major investigation late in the 1980's, several companies worldwide have tried to develop investment casting technology for second generation gamma alloys. For the last few years, the gamma casting technology has advanced considerably through solving various problems such as cracking, hot tearing, surface connected porosity, filling and dimensional accuracy. In addition, much effort now appears to be directed toward establishing low cost, consistent manufacturing processes which incorporate alloy composition and its variations, materials properties, casting conditions and parameters, fillability, HIP'ing and final microstructures of interest. [14].

However, as-cast/HIP'ed Ti-48Al-2Cr-2Nb is unacceptably low in ductility and strength for many applications, due mainly to the coarse and nonuniform cast lamellar microstructure, which is not readily removed by HIP'ing. Empirical efforts have been made to control, through annealing treatments, the lamellar structure into finer mixtures of gamma grains and residual lamellar regions which are called "casting duplex" microstructures of about 100-200µm grain size. The casting duplex form of Ti-47Al-2Cr-2Nb exhibits a reasonable balance of properties (though relatively low levels), and has recently been demonstrated as a viable engineering material through rigorous engine tests. During this period, investigations to refine cast microstructures have been made, resulting in the development of cast XD alloys first at Howmet in 1990. Two XD alloys, Ti-(45, 47)Al-2Mn-2Nb-0.8vol%TiB2, have been tested worldwide and appear to be establishing themselves as engineering alloys. The inoculation ability of boron in cast alloys was also used in Japan (IHI) and Germany (GKSS) to develop cast alloys, Ti-47Al-1.6Fe-1.4V-2.4B and Ti-47Al-3.5(Nb, Cr, Mn)-0.8(B, Si), respectively. [2-5]

Databases for the second-generation cast alloys are being established through extensive property evaluation on a few fixed processing-microstructure conditions. Most of the properties measured at temperatures up to 760°C appear to be comparable to, or better than, when adjusted for density, those of the counterpart Ni-base superalloys which they are to be substituted for. Fatigue crack growth, impact resistance and ductility are of concern, and appropriate measures may be needed in design strategy to accommodate such deficiencies.

### Casting Alloys

Various gamma components for turbine engines have been identified for rotational parts such as low pressure turbine (LPT), high pressure compressor (HPC) blades, and high pressure turbine (HPT) blade cover plates, and stationary parts, such as transition duct beams, vanes, swirlers, various cases, and nozzle flaps and tiles.

For the past few years, the databases and damage tolerance of various gamma alloys have been assessed for some of the identified components, through various qualification tests including bench tests, rig tests, and engine tests, by several companies including GE, P&W, MTU, Rolls-Royce, and IHI. Perhaps, the most significant qualification tests were the rigorous engine tests conducted in 1993 and 1994 by GE on a full set wheel of 98 LPT cast gamma blades made of Ti-47Al-2Cr-2Nb. The two successful engine tests including over 1500 simulated flight cycles were a milestone for gamma and planted a definite, though not totally certain, confidence on the material in the gamma TiAl community as well as designers. Through these and other tests, casting gamma alloys are proving to be technologically sound materials and, with some design modifications pertinent to each component, can replace nickel based superalloys in use for selected engine components. Accelerated uses of gamma alloys in replacing the current materials will be warranted when many uncertainties about the performance in the field are cleared or answered and low cost manufacturing processes are demonstrated for important types of components. [3, 14, 15].

Cast TiAl alloys are also intended for use in automotive engine parts such as turbochargers and valves. Recent engine tests show that cast TiAl turbocharger rotors exhibit better acceleration response and higher maximum rotational speed than its counterpart, Inconel rotor. There are some concerns in accepting a TiAl turbocharger, such as low ductility at room temperature and high temperature (above 800°C) oxidation resistance. Nevertheless, its application appears to be imminent, especially, in large diesel engines. Exhaust engine valves appear to be an ideal application for gamma alloys which are expected to replace the current valves made of steel and/or nickel-base (Inconel) alloys. The properties of gamma alloys in most microstructural forms well exceed most of the property requirements, as was demonstrated through series of extensive qualification engine tests conducted at GM recently. The remaining barrier appears to be development of a low-cost, high volume manufacturing method. At present, casting and perhaps reactive sintering are the two most important production methods, and intensive production and alloy modification efforts to reduce the cost is underway worldwide. [3, 14, 15].

### Fundamental Advances

While casting gamma technology has been progressing for producing components, the advances in understanding of many fundamental aspects of the alloys has been impressive. The mid-section of Ti-Al binary phase diagram has been established, and some ternary diagram work has progressed. The sequence of transformations involving  $\alpha$  decomposition have been qualitatively understood. For the alloy compositions of engineering importance [Ti-(45-48)Al base], decomposition takes place in several paths, depending on cooling rate and method, yielding lamellar structures under relatively slow cooling, "feathery"- type structures under air cooling and massive gamma when water-quenched. Investigations have concentrated on lamellar structures for formation mechanism, growth kinetics and alloy design. Extensive investigations of the deformation behavior of unidirectional lamellar material has been conducted mainly at Kyoto and Osaka Universities to establish the deformation anisotropy, which is extraordinary. Fine details and various mechanical behavior are under continuos investigation worldwide. Advanced understanding of the lamellar structure will be crucial for designing optimal lamellar base microstructures. [4-9, 12-14].

Based on knowledge of the phase relations and transformations, progress has been made for the last several years, in controlling and understanding of microstructures developed in wrought-processed alloys at Wright-Patterson as well as other institutes. Four different types of standard

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microstructures were identified: near-gamma, duplex, nearly-lamellar (NL) and fully-lamellar (FL) types. The first two are fine gamma grain based (<60µm), the last two are lamellar based, and FL material in general is large-grained (>350µm). Gamma alloys show the 'so-called' ductility-toughness inverse relation at temperatures below the BDT temperatures. Fine grain microstructures yield improved tensile properties but low fracture toughness, and the reverse is true for large grain FL material. This relation is explained by correlating grain size (GS), pileup-dislocation-density, and the deformation anisotropy of the lamellar structure. However, a critical GS appears to exist, above which further increases of toughness ceases. The anisotropy also appears to be responsible for the abnormally high strength gain with decreasing grain size, as well as the improvements of strength and toughness with decreasing lamellar spacing. Creep resistance is higher for FL than duplex microstructures and appears to increase with grain size. Additions such as Si, C, N, Ta and W appear to improve the creep resistance, although each mechanism is known only qualitatively. High cycle fatigue resistance is excellent at least up to 800°C, but becomes oxidation limited in general at higher temperatures. Relatively low fracture toughness and very fast fatigue crack growth rates are of concern because the material's life will then be limited in the presence of relatively small existing or created defects/flaws. Nevertheless, both properties are improved in FL material and with increasing grain size. [3-5, 12, 14, 15].

### **Wrought Processing**

It is estimated, considering the inverse ductility-toughness relation and the beneficial effects of lamellar structures on fracture toughness, creep, high temperature fatigue properties and fatigue crack growth resistance, that FL material having a grain size roughly in the range from 50-400  $\mu m$  should show improved balance in properties. The exact magnitude should be a function of property requirements, component configurations and dimensions (thickness) and processing method.

Since such a controlled FL grain size is difficult to produce in Ti-48Al-2Cr-2Nb, and since alloys containing large amounts of boron (such as XD alloys) are not suitable as wrought alloys, extensive investigations have been conducted to design refined lamellar structures in wrought alloys. Such designed materials have been obtained in relatively thin sections: by adding small amounts (<0.3 at%) of boron (yielding TMT lamellar); through appropriate high temperature extrusion (producing TMP lamellar); or by appropriate alloy modifications which widen/lower the high temperature ( $\alpha+\beta$ ) phase field (resulting in refined FL). Methods to control lamellar spacing were also developed utilizing the lamellar formation mechanism and growth kinetics. Fundamentally, we are just beginning to sufficiently understand the essential aspects of designing lamellar structures. [3, 4, 14]

Almost at the same time, investigations of workability and texture development during hot working have been extensively investigated in USA, Japan and Germany. On the basis of the fundamental understanding and using concurrently developed process modeling, advances in process development have been realized for wrought processing in areas such as primary processing, secondary processing, and component forming. Ingot conversion through isothermal forging, extrusion and multistep processing has been commonly practiced with and without homogenization treatments on a small scale. Conversion of large ingots (over 250kg) is now possible through multistep processing, although more details have yet to be answered before production scale practices can be implemented. Ingot breakdown by non-isothermal forging has been shown to be feasible using a canned workpiece.

Pack-rolling technology has been advanced using both forged plates as well as prealloyed powder compacts, with sound sheet of 800x300x1.5mm currently produced. The availability of the size and microstructural homogeneity of starting plates appears to limit production of larger sheet. In general, hot-worked gamma material is highly formable, isothermally, at temperatures as low as 900°C. Prototype blades having twisted air foils have been successfully forged isothermally, and rolled sheet has been superplastically formed into various complex-shape parts. Forming by hot-die forging and high rate extrusion, however, is a hurdle for gamma to overcome if wrought gamma components are to be produced cost-effectively. Recent trials of automotive valve extrusion in current production facilities, though limited to canned/insulated material, indicate that such high rate forming of gamma may eventually be feasible if optimum processing conditions and parameters are identified on preconditioned material. [6-10, 14, 15]

Production of gamma ingots has been practiced using various melting and casting methods including induction skull melting (ISM), vacuum arc remelting (VAR), plasma arc melting (PAM) and VIM (vacuum induction melting). Ingots having 30cm diameter are routinely produced by the VAR technique. Ingots with 36 cm diameter and weighing more than 250kg have been produced using

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VAR and PAM techniques, and production of larger ingots by PAM appears feasible. The main concerns in scaling-up are cracking, control of chemistry (especially, aluminum level), and compositional variations along the ingot length. In addition, methods to produce ingots having more refined and uniform cast structures are yet to be developed. [15]

### Alloy Design

As the importance and necessity of the properties pertinent to specific components is recognized, increasing efforts have been made for the last couple of years to develop specific materials through microstructure control and alloy modification. In this effort focused more on wrought alloys, the thrust was to develop lamellar-based structures which are fine enough for specific component thicknesses and coarse enough to retain the most desirable properties. The important microstructures developed from the effort include TMT, RFL and TMP microstructures. The most dramatic improvements were observed in TMP materials, with strength levels reaching as high as 1000MPa at RT and more than 500MPa at 1000°C.

Nevertheless, considerably more work has to be done in various aspects such as understanding the formation mechanisms, thermal and mechanical stability, process control including heat treatment cycles, establishing data bases, property evaluation, and characterization of damage tolerance. It will also be important to raise the property levels (creep and strength) by adding small amounts of C, Si, N or O, without affecting other properties. With the above modifications, the component-specific gamma materials are expected to exhibit improved balances of properties and/or increased use temperatures by 50-100°C. These types of improvements appear to be possible in cast alloys by alloy modifications (recent results at GE) and refining coarse cast lamellar grains into fine lamellar grains by novel heat treatment cycles (results at Wright-Patterson). [3, 4, 14].

Further increases in use temperature of gamma material to above 850°C could be very profitable in the future and may be accomplished through the development of novel processes, new alloys and effective surface treatments (including protection) methods. The novel processing methods, which are under exploration, are aimed at producing material having aligned lamellar structures by directional solidification (DS) of columnar grains and/or directional extrusion (DE) of lamellar grains. These methods have yet to show their engineering feasibility to produce the intended microstructures and then the resulting microstructures/materials must be shown to demonstrate their expected higher temperature capabilities as well as the damage tolerance comparable to those of current gamma alloys. In the end, however, it appears inevitable to protect the surface of any gamma alloys if they are to be used at temperatures above 800-850°C. Several preliminary or developmental efforts in this area, however, suggest that this is most challenging and will not happen quickly [14].

Increases in both oxidation resistance and higher temperature strength levels may require drastic departures in composition from the current gamma alloys. One example can be alloys containing large amounts of Niobium. Development of such new alloys requires prolonged research efforts which nevertheless are very much worthwhile.

### Summary and Future

Gamma alloys are emerging as important engineering materials. These alloys are a rare example of how the research community, developers, producers, and users can work closely to speed up and steer a materials technology in the right directions. The current alloys, basically cast alloys, developed under such remarkable collaboration meet property requirements of selected turbine, as well as automotive, engine components. With the development of appropriate design methodologies and cost effective manufacturing methods, these alloys are certain to be implemented for selected applications in the near future. In the meantime, the research community faces (new) challenges: designing engineering wrought alloys, controlling new and/or improved processing/microstructures for immediate, specific applications, and development of new materials/alloys for higher performance and temperature applications. This surely will be a long road, but should be rewarding as their past effort. There is increasing evidence that the research community is no longer naive or close-minded as too many are quick to assume. When both the research and producer/user communities work closely together, posturing to learn from each other, the gamma technology will be advanced even faster, finding more diversified application areas which will benefit all communities.

### References

- 1. M.J. Blackburn and M.P. Smith, AFWAL Technical Report, 80-4175 and 82-4086(1980-82).
- 2. H.A. Lipsitt, Advanced High-Temperature Alloys (ASM, 1986), pp. 157-164.
- 3. Y-W. Kim, JOM, 14 (7) (1989), pp. 24-30 and 46 (7) (1994), pp. 30-40.
- 4. Y-W. Kim and D.M. Dimiduk, JOM, 43 (8) (1991), pp. 40-47.
- 5. M. Yamaguchi and H. Inui, Structural Intermetallics (Warrendale, Pa, TMS), pp. 127-142.
- 6-9 High Temperature Ordered Intermetallic Alloys II-V (1989-1995), (Pittsburgh, Pa, MRS).
- 10. Microstructure/Property relationships in Titanium Aluminides and Alloys, TMS proceedings (Warrendale, Pa, TMS, 1990).
- 11. High Temperature Aluminides \$ Intermetallics (Warrendale, Pa, TMS, 1990).
- 12. Structural Intermetallics (Warrendale, Pa, TMS, 1993).

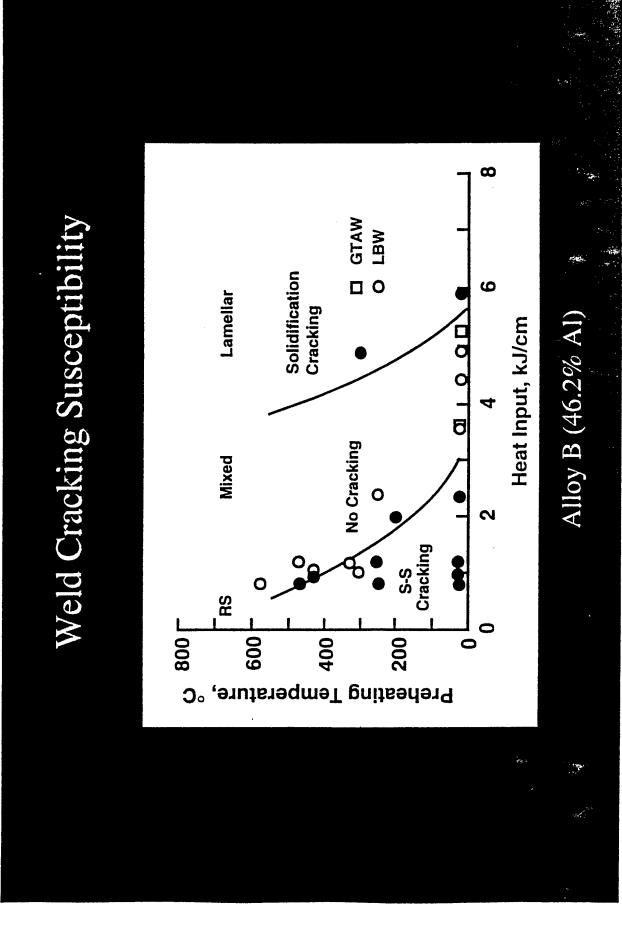
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- 13. Intermetallic Compounds, Proc. 3rd Japan International SAMPE Symp. (Chiba, Japan, 1993).
- 14. Proc. on International Symp. on *Gamma Titanium Aluminides*, ed. Y-W. Kim, R. Wagner and M.Yamaguchi (Warrendale, Pa, TMS) 1995
- 15. Communications: C. Austin (GEAE), M.J. Blackburn (P&W), P. Bowen (IRC), K. Chan ISWRII

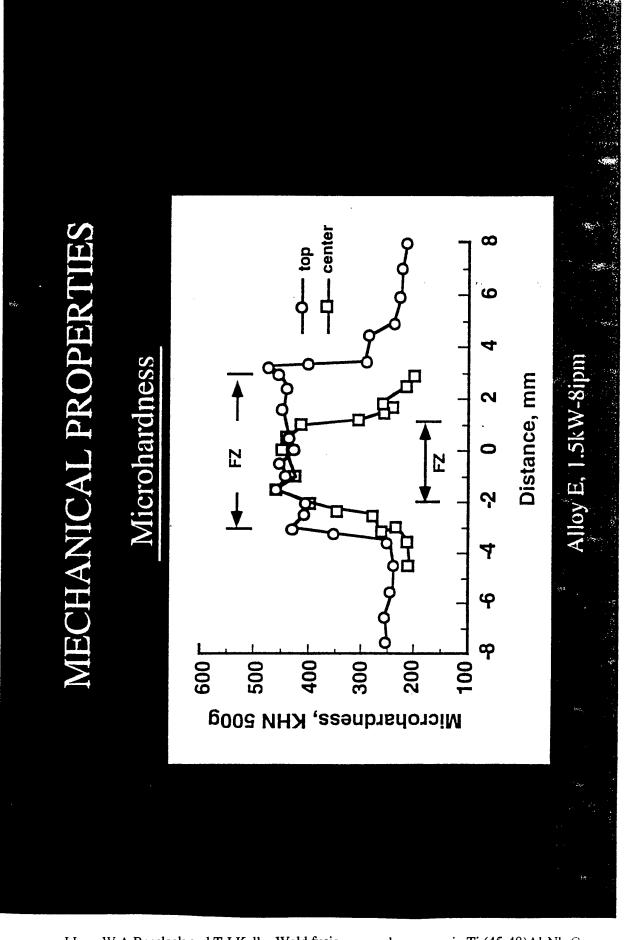
[SwRI],
H. Clemens (Plansee), D. Davidson (P&W), D. Dimiduk (Wright Lab.), D. Furrer (Ladish), S. Hartfield-Wünsch (GM), H. Huang (GE), W. Konkel (W-G), D. Larsen, Jr. (Howmet), J. Larsen (Wright Lab.), C. T. Liu (ORNL), P. Bowen (IRC), S. Naka (ONERA), Y. G. Nakagawa (IHI), S. Ram (PCC), S. Reed (Duriron), S. Schwenker (Wright Lab.), L. Semiatin (Wright Lab.), D. Shih (MD), W. Smarsley (MTU), R. Wagner (GKSS), and M. Yamaguchi (Kyoto U.).

### Overview of joining gamma alloys

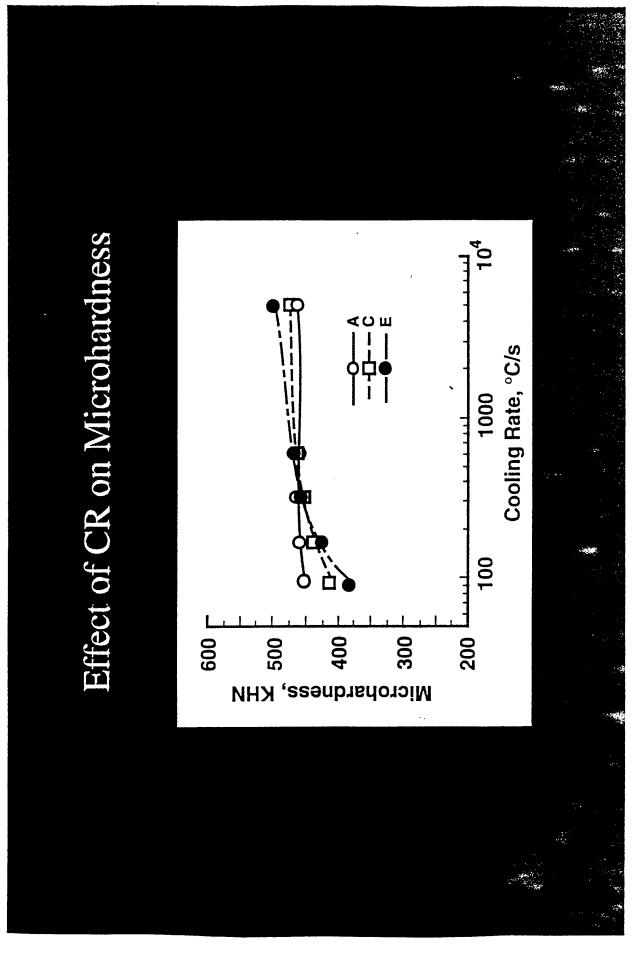
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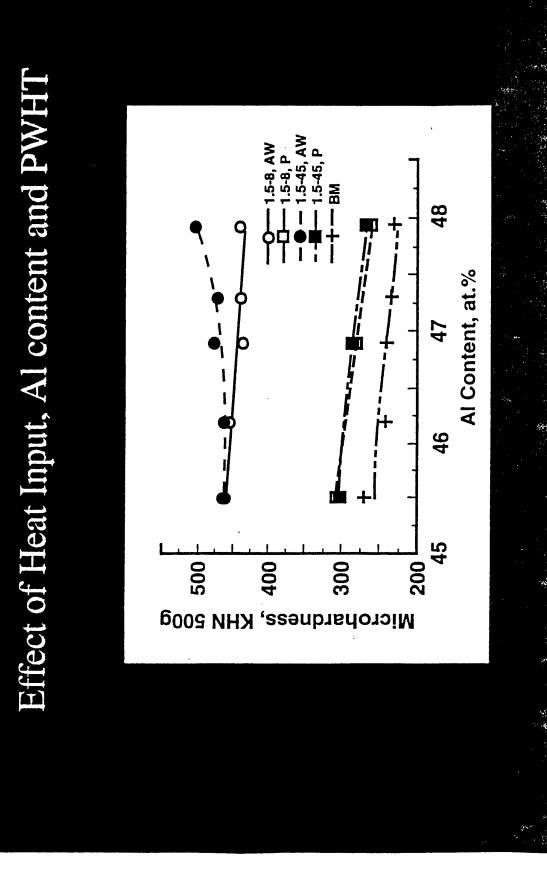
J Lee, W A Baeslack and T J Kelly: Weld fusion zone phenomena in Ti (45-48)Al-Nb-Cr  $\gamma$  titanium aluminides, submitted to Welding Journal.



J Lee, W A Baeslack and T J Kelly: Weld fusion zone phenomena in Ti (45-48)Al-Nb-Cr  $\gamma$  titanium aluminides, submitted to Welding Journal.



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#### CONCLUSIONS

- Gamma titanium aluminides were found to be susceptible along centerline and cracking susceptibility was determined to solidification cracking. Solidification cracking occurred by fusion zone morphology.
- as heat Solidification cracking susceptibility increased input and Al content increased.
- Solid-state cracking susceptibility decreased as cooling rate decreased and Al content increased. Process windows for crack-free sound LB welds were established in this study

#### CONCLUSIONS

- Microhardness of the as-welded fusion zone was much higher than the base material but weld cooling rate did not influence microhardness of fusion zone significantly.
- PWHT reduced microhardness of the fusion zone to the level of the base material.
- it was apparent that PWHT increased the fusion zone ducti Based on fractography,

### MICROSTRUCTURE EVOLUTION IN THE ENOZ NOISIE

• FZ with fast cooling rates:

Low Al Alloy; Incipient Lamellar +  $\alpha_2$  Grains +(B2 phase) High Al Alloy; Incipient Lamellar +  $\gamma$  Grains +(B2 phase)

FZ with slow cooling rates:

High Al Alloy; Lamellar + Blocky γ Grains +(B2 phase) Low Al Alloy; Fully Lamellar + Fine GB  $\gamma$  +(B2 phase)

Lamellar spacing increase with H.I.

Dendrite and columnar grain size increase with H.

# SOLIDIFICATION OF GAMMA TITANIUM ALUMINIDES

- Fusion zone: cored dendritic structure
- duplex solidification( $\alpha+\beta$ ) with increasing Al content Primary solidification mode change from single 10 BCC \(\beta\) solidification and/or CR phase
- Solute redistribution during solidification:
- -Al segregation to interdendritic regions
- -Cr enrichment and Al depletion on dendrite cores

#### WELD CRACKING SUSCEPTIBILITY OF GAMMA TITANIUM ALUMINIDES

- Susceptible to both solidification cracking and solid-state cracking.
- Solidification crack: parallel to the welding direction

propagate along centerline

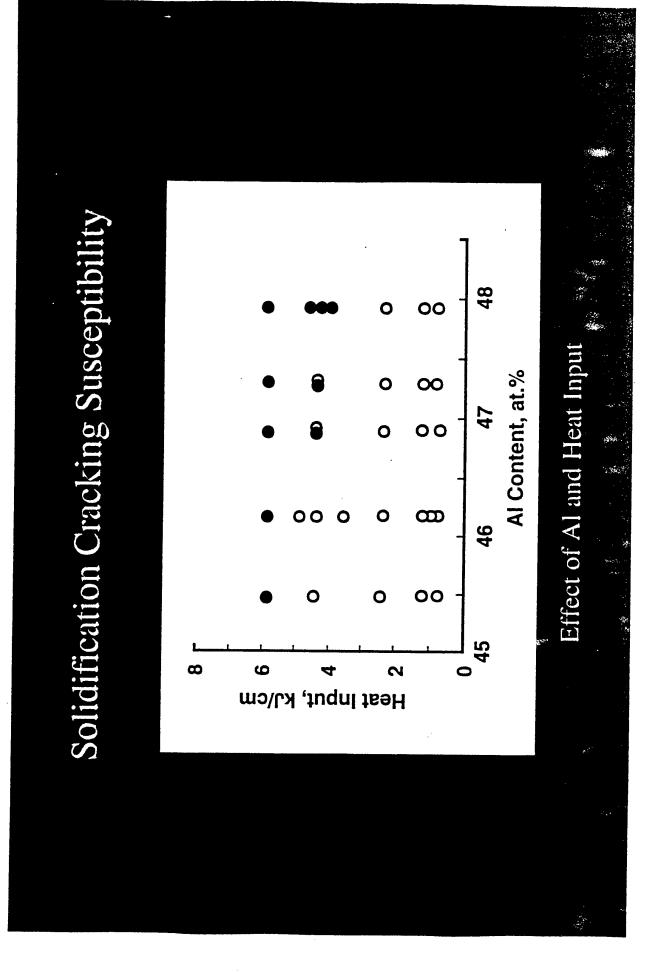
Solid-state crack: perpendicular to the welding direction

- As Al content and H.I. increased, solidification cracking susceptibility increased while solid-state cracking susceptibility propagate along alpha-two phase preferentially decreased.
- Process windows for sound LB welds were established

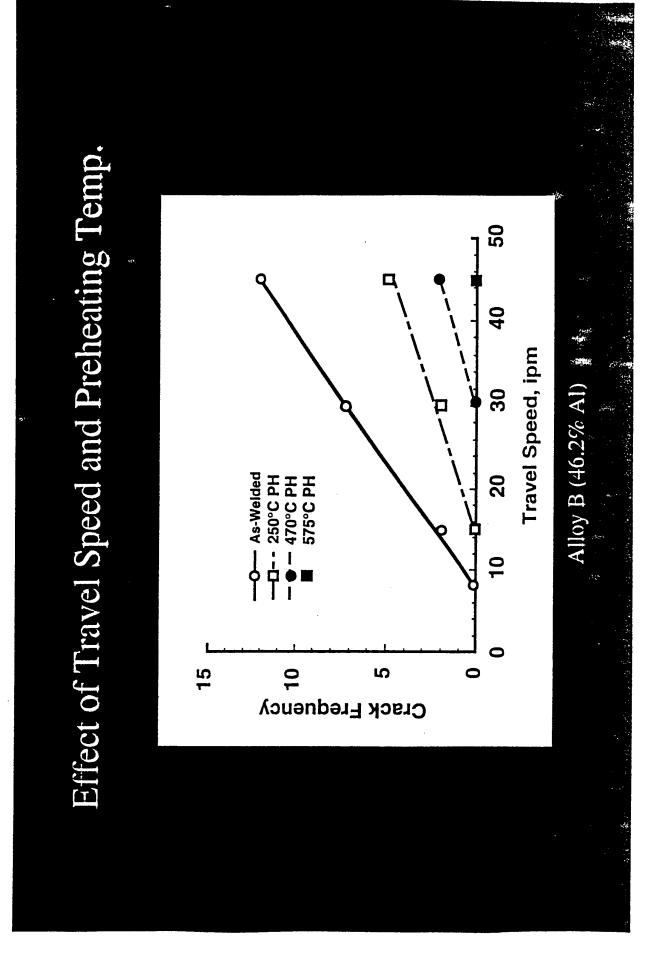
# EXPERIMENTAL PROCEDURE

#### Materials

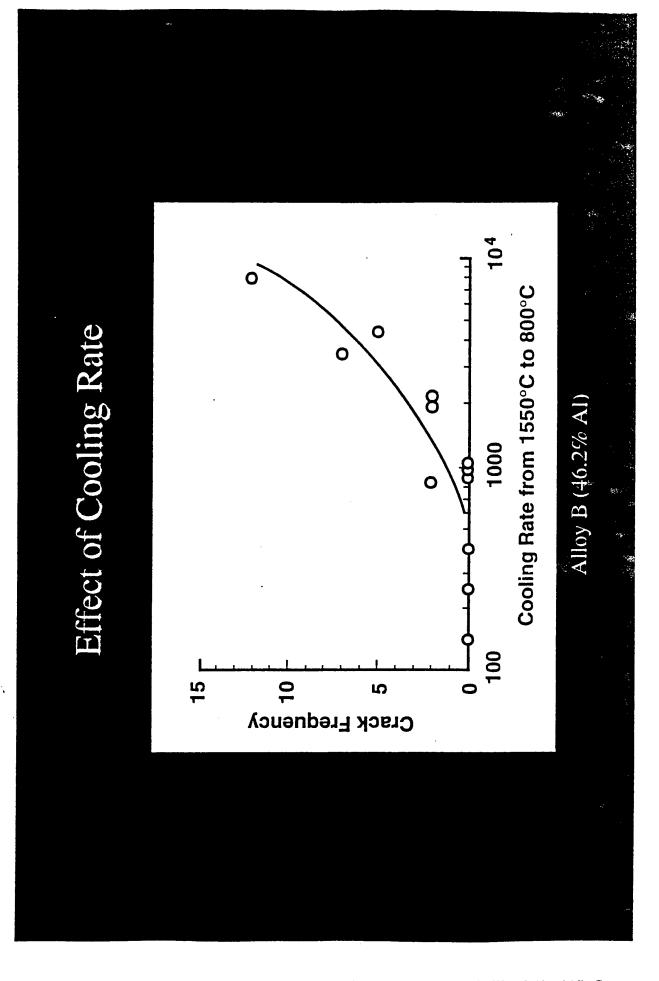
HT	Ti Al	Aį	Nb	Cr	Si	Fe	0
A	50.6	50.6 45.5	1.9	1.8	.028	.028	.221
B	49.8	46.2	2.0	1.7	.04	.04	.13
C	49.4 46.9	46.9	1.9	1.5	.03	.03	.14
D	D 48.6 47.3	47.3	1.9	1.9	.03	.03	.14
田	48.4 47.9		1.9	1.5	.03	.03	.15



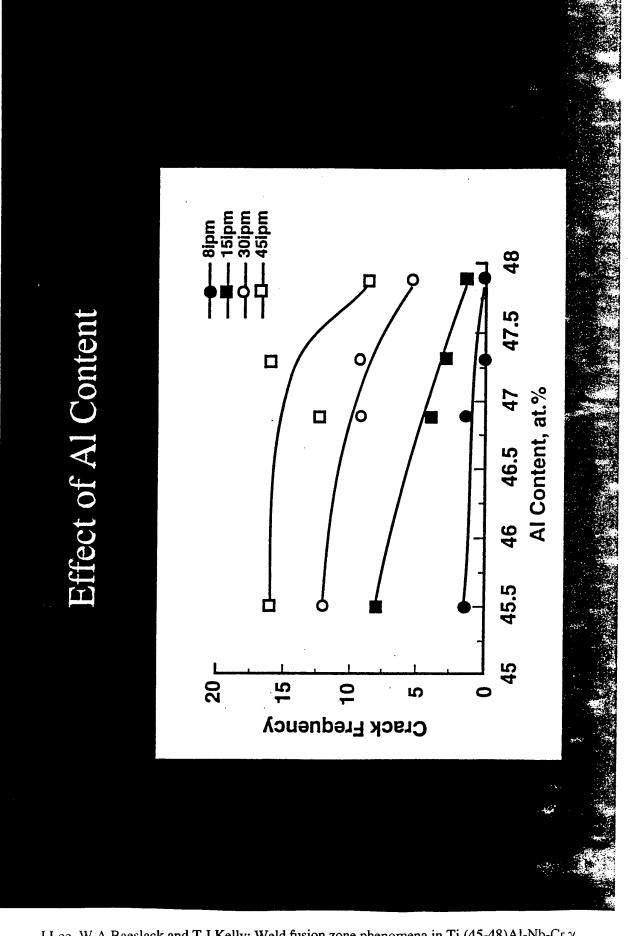
J Lee, W A Baeslack and T J Kelly: Weld fusion zone phenomena in Ti (45-48)Al-Nb-Cr  $\gamma$  titanium aluminides, submitted to Welding Journal.



J Lee, W A Baeslack and T J Kelly: Weld fusion zone phenomena in Ti (45-48) Al-Nb-Cr  $\gamma$  titanium aluminides, submitted to Welding Journal.

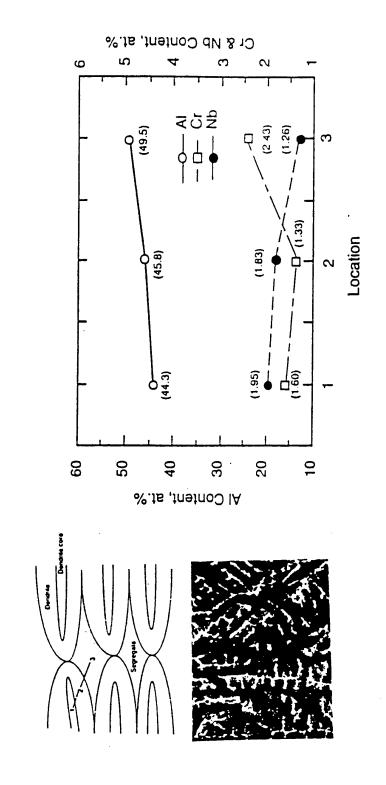


J Lee, W A Baeslack and T J Kelly: Weld fusion zone phenomena in Ti (45-48)Al-Nb-Cr  $\gamma$  titanium aluminides, submitted to Welding Journal.

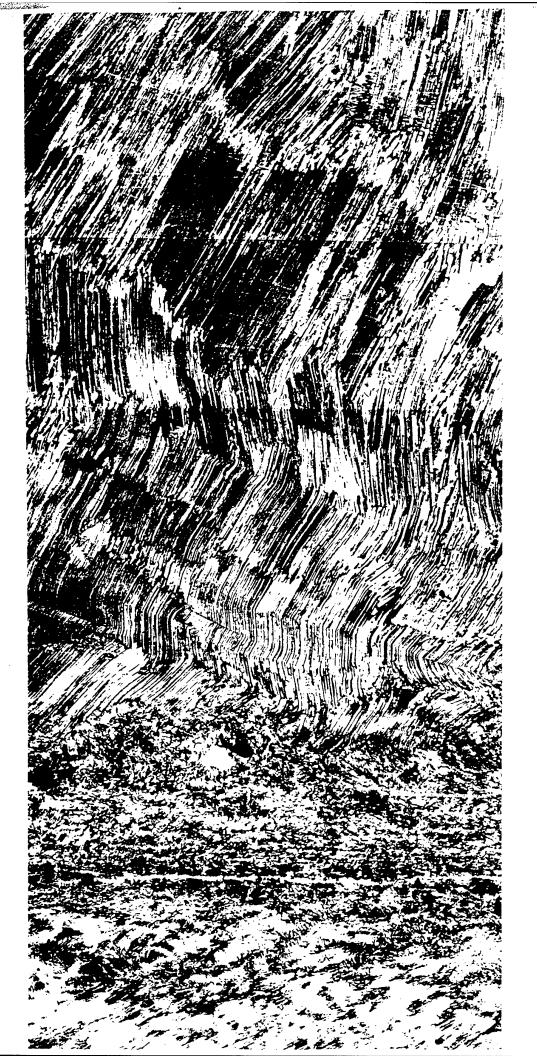


J Lee, W A Baeslack and T J Kelly: Weld fusion zone phenomena in Ti (45-48)Al-Nb-Cr  $\gamma$  titanium aluminides, submitted to Welding Journal.

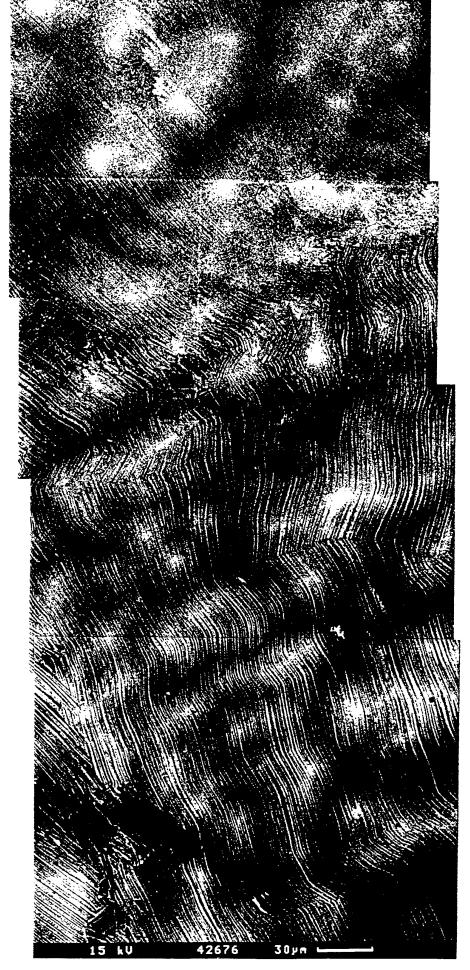
# Partitioning of Alloying Elements during



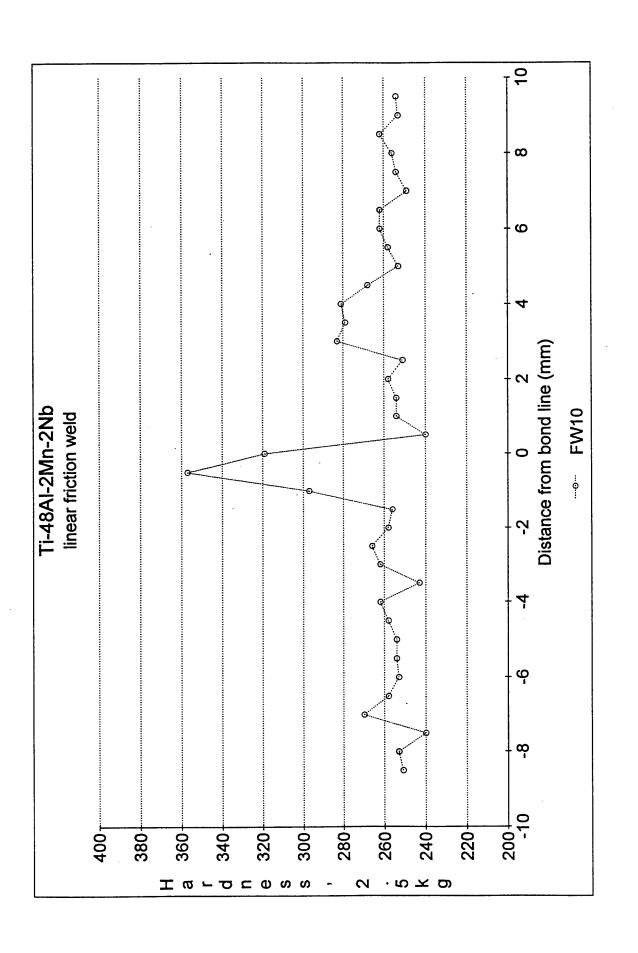
Alloy E, 1.5kW-8ipm

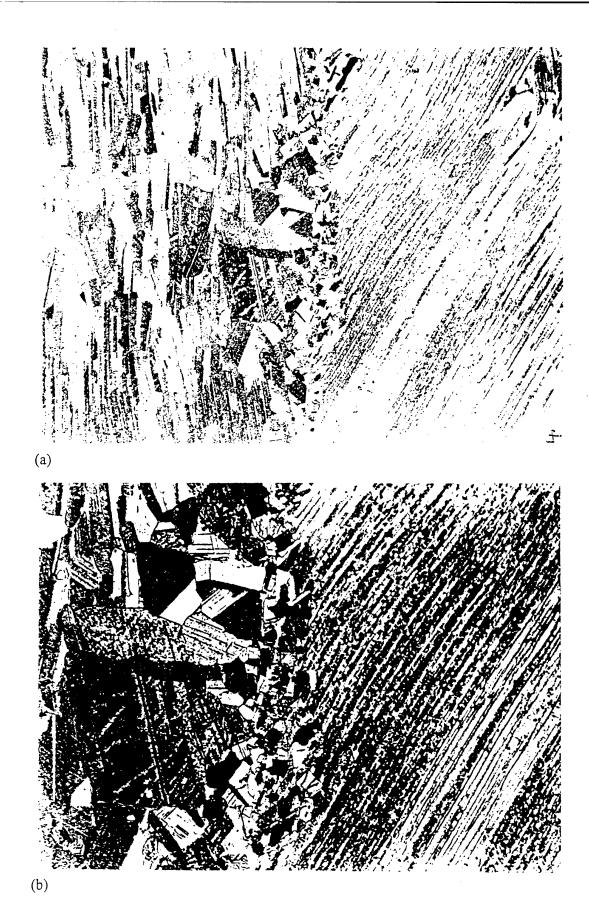


Linear friction weld in 10mm Ti-48Al-2Mn-2Nb, FW10, x100. AG2210 (top) - AG2213



Backscattered electron image of HAZ microstructure of linear friction weld FW10 in Ti-48Al-2Mn-2Nb casting, in unetched condition, x500. Negs 42676,-77,-78,-80

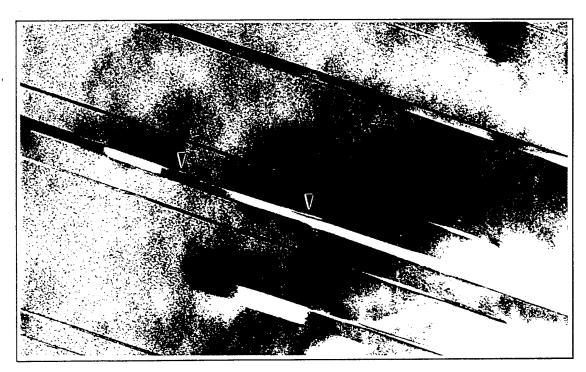




Electron beam diffusion bond in Ti-48Al-2Mn-2Nb, W81, 1200°C, 20MPa. (a) x500, AG2021 (b) x1000, AG2022

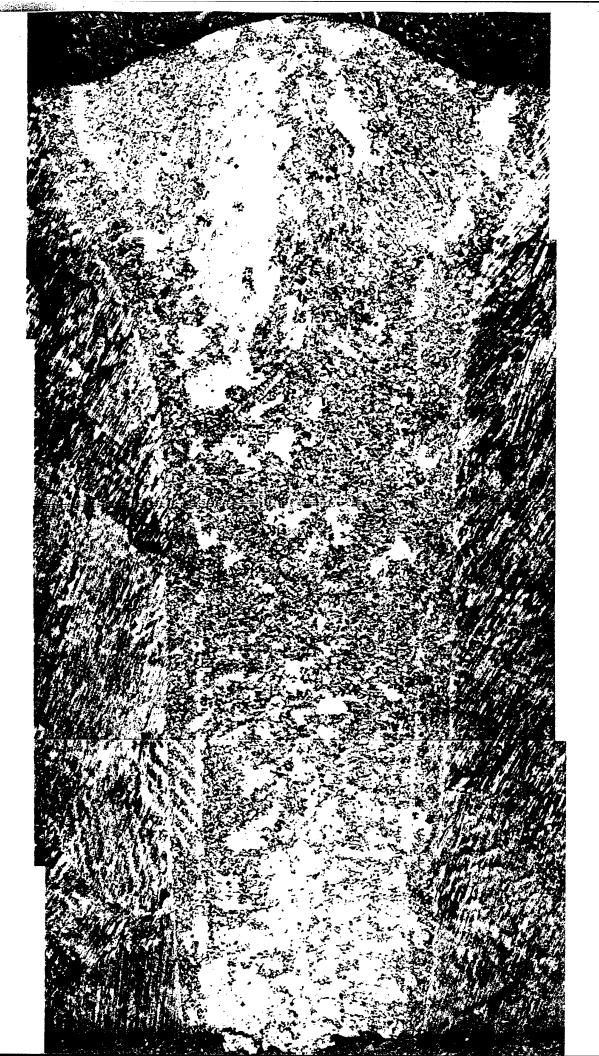


 ${\bf 150~nm} \\ Bright field electron micrograph showing fine laths in 'retained~\alpha' regions of electron beam welded Ti-48at.%Al-2at.%Mn-2at.%Nb.~Arrows indicate possible growth steps on the laths$ 

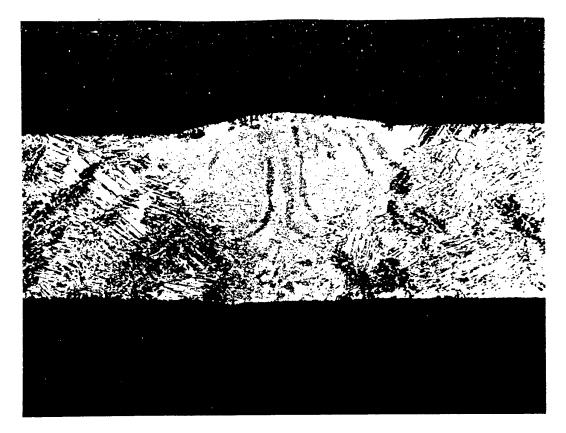


50 nm

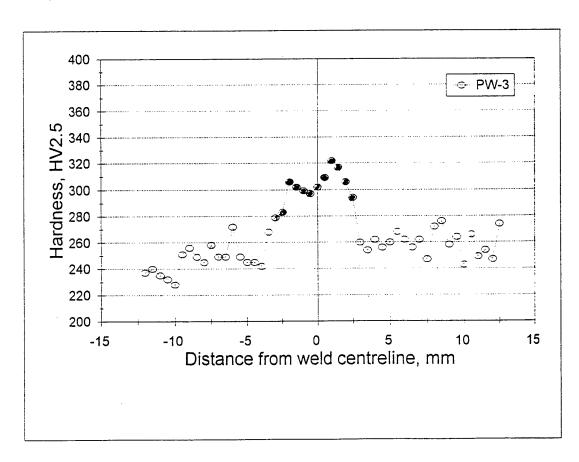
Bright field electron micrograph showing fine laths in 'retained  $\alpha$ ' regions of electron beam welded Ti-48at.%Al-2at.%Mn-2at.%Nb. Arrows indicate possible growth steps on the laths

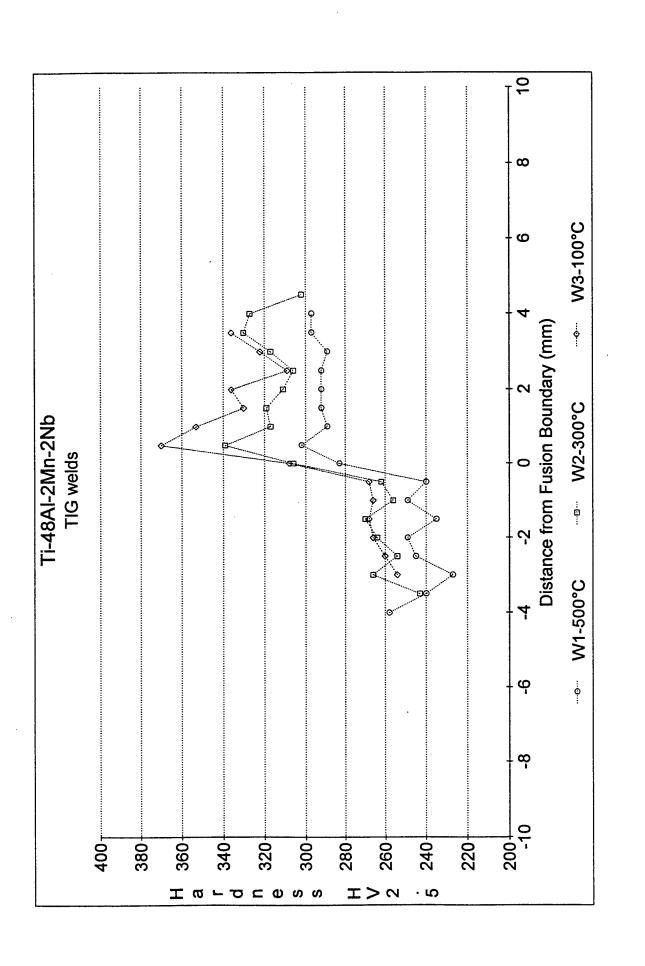


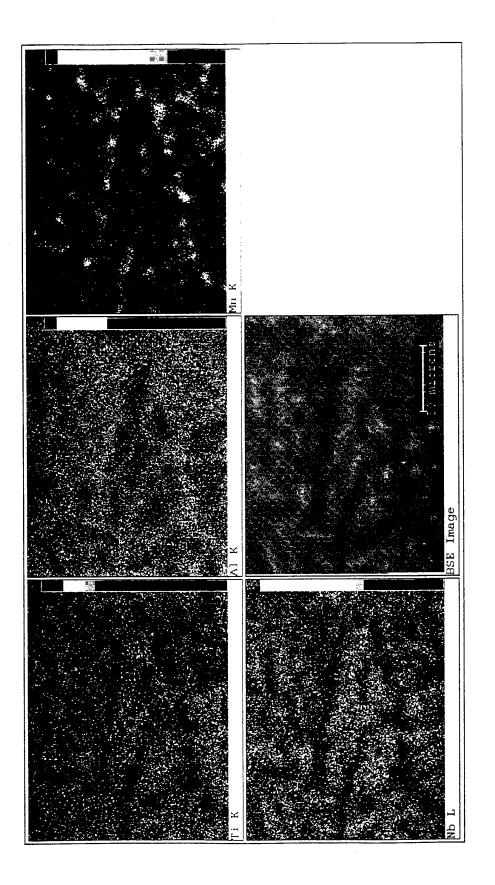
Electron beam weld in 5mm Ti-48Al-2Mn-2Nb, no preheat, x50. (Birmingham University)



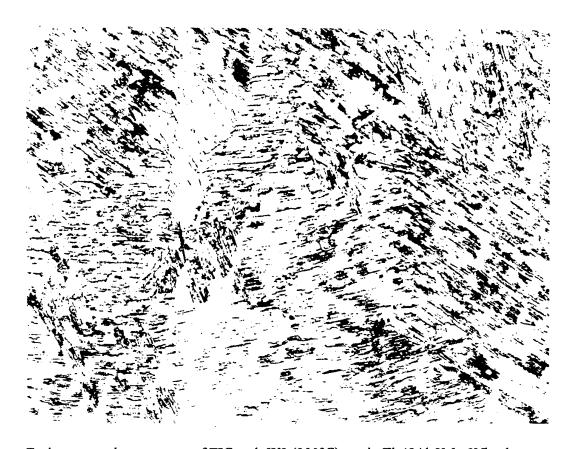
Autogenous keyhole plasma weld in Ti-48Al-2Mn-2Nb casting, x10



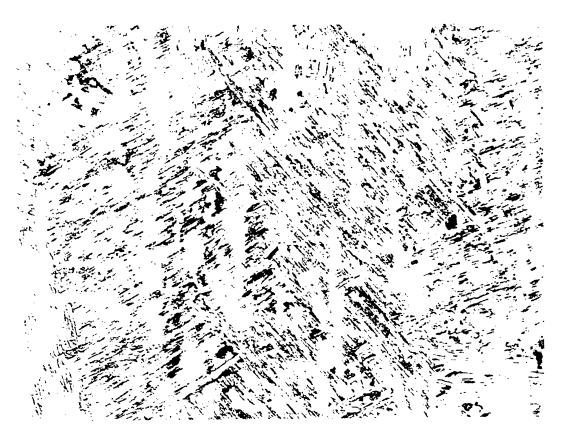




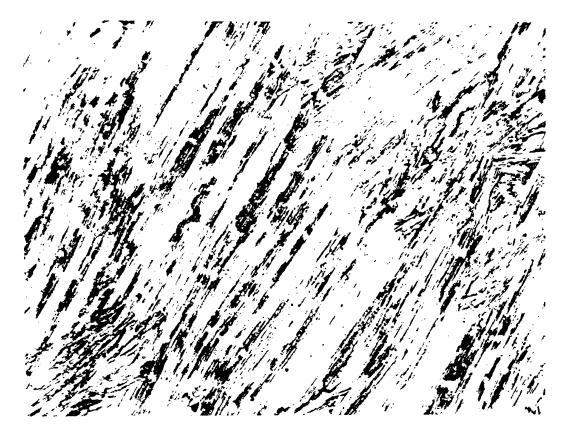
Element distributions in TIG weld W2 in Ti-48Al-2Mn-2Nb



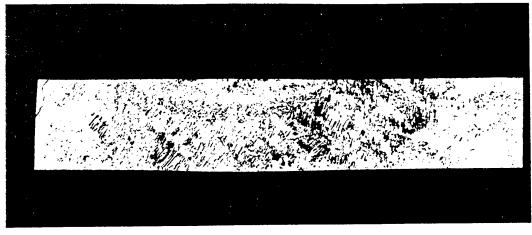
Fusion zone microstructure of TIG melt W2 (300°C) run in Ti-48Al-2Mn-2Nb, close to surface. x500. AG1620



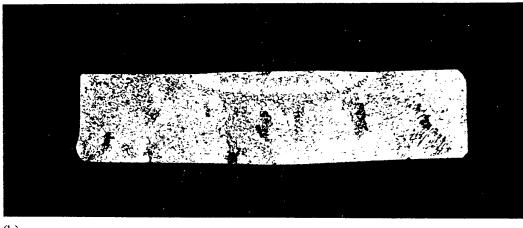
Fusion zone microstructure of TIG melt W3 (100°C) run in Ti-48Al-2Mn-2Nb, close to surface. x500. AG1621



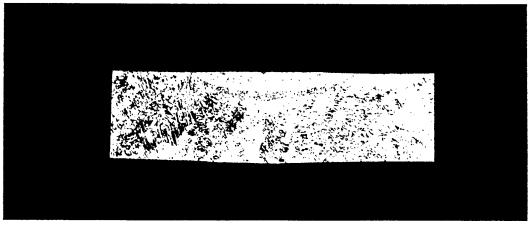
Fusion zone microstructure of TIG melt W1 (500°C) run in Ti-48Al-2Mn-2Nb, close to surface. x500. AG1619



(a)



(b)



(c)

TIG melt runs in Ti-48Al-2Mn-2Nb, x5 (a) W1, 500°C, AG1630. (b) W2, 300°C, AG1629. (c) W3, 100°C, AG1628.